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LABORATORY MANUAL

OF

ELEMENTARY CHEMISTRY

BY

J. MONTGOMERY, PH.D.

AND

R. B. SMITH

ANN ARBOR HIGH SCHOOL.

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1897.

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PREFACE.

This little book is intended for a laboratory guide and should be used in connection with a good text-book, or, a course of lectures fully illustrated by experiments.

It calls for no work that may not be done by the average high school pupil. As a matter of fact nearly all of these experiments have been performed by such pupils many times over; one of the authors having used most of them for twelve or fifteen years.

The explanations may appear to be unnecessarily minute. Experience teaches, however, that it is almost impossible to be too careful with the beginner.

The attempt has been made to throw the pupil more and more upon his own resources as he becomes more familiar with chemical manipulation.

The experiments on the metals are intended to acquaint the pupil with the properties of important metals and some of their compounds. The tables are not intended for complete analysis, for which they are scarcely fitted, but rather, to be the basis of a series of exercises which will lead the pupil, in some measure, to comprehend the methods of analytical chemistry. Indeed, the authors doubt the propriety of attempting to teach chemical analysis in high schools, but, experience has shown them that some such work has an excellent effect in creating interest and enthusiasm.

A few problems in quantitative analysis have been inserted to give the learner an idea of accurate work.

In most cases a few of the commoner metals and their important compounds will be all that can be studied. However, the extent of this kind of work will depend upon the time and laboratory facilities at the disposal of the instructor.

A lack of uniformity of treatment will be observed. This is, in a sense, intentional. The arrangement of matter and the insertion or omission of formulæ, etc., has all been made to depend upon what seemed best for the pupil.

The sources of materials used will be recognized by any one somewhat familiar with chemical literature. The special form of some of the experiments was devised long before they appeared in any text-book.

The figures and tables have been selected and modified to suit our purposes and are found in many text-books published during the last fifteen or twenty years. For the tables in analysis, however, the works of Prescott and Johnson, Will, and Fresenius have been most frequently consulted. In some of these but slight modification has been made.

Frequently the atomic weight to the nearest whole number, or large decimal, only, has been given. The accurate determinations will be found in the table.

Rules to guide the pupil in the laboratory are sometimes inserted in manuals like this. We have omitted everything of this kind, believing that the competent teacher can prepare directions that will suit his purposes better than any one else.

We wish to acknowledge our indebtedness to many friends for suggestions, proof reading and other assistance, so kindly rendered, while the press work was in progress. We shall be glad to receive suggestions and corrections from any and all, that errors may be eliminated from future editions should there be such.

Hoping that our book may be helpful to teacher and pupil, we commend it to all educators who believe that contact with the thing is the only way to gain real scientific knowledge.

ANN ARBOR, MICH., 1897.

TABLE OF COMMON ELEMENTS.

Name.	Symbol.	H=1.	O=16	Discovery.	Discoverer.
Aluminum.....	Al	26.91	27.11	1827	Wöhler
Antimony.....	Sb	119.52	120.43	15th century	Basil Valentine
Argon.....	A	?	?	1894	Rayleigh and Ramsay
Arsenic.....	As	74.44	75.10	1694	Schröder
Barium.....	Ba	136.39	137.43	1808	Davy
Bismuth.....	Bi	206.54	208.11	15th century	Valentine
Boron..	B	10.86	10.95	1808	Gay Lussac and Thenard
Bromin.....	Br	79.34	79.95	1826	Balard
Cadmium.....	Cd	111.10	111.95	1817	Stromeyer
Calcium.....	Ca	39.76	40.07	1808	Davy
Carbon.....	C	11.92	12.01	Ancient	.....
Chlorin.....	Cl	35.18	35.45	1774	Scheele
Chromium.....	Cr	51.74	52.14	1797	Vauquelin
Cobalt	Co	58.49	58.93	1742	Brandt
Copper.....	Cu	63.12	63.60	Ancient	.....
Fluorin.....	F	18.91	19.06	1886	Moissan
Gold.....	Au	195.74	197.23	Ancient	.....
Helium.....	He	?	?	1895	Ramsay
Hydrogen	H	1.00	1.008	1766	Cavendish
Iodin.....	I	125.89	126.85	1811	Courtois
Iron	Fe	56.60	56.02	Ancient	.....
Lead...	Pb	205.36	206.92	Ancient	.....
Magnesium...	Mg	24.10	24.28	1808	Davy
Manganese ...	Mn	54.57	54.99	1774	Gahn
Mercury.....	Hg	198.49	200.06	Ancient	.....
Nickel	Ni	58.24	58.69	1750	Cronstedt
Nitrogen	N	13.93	14.04	1772	Rutherford
Oxygen	O	15.88	16.00	1774	Priestly
Phosphorus ...	P	30.79	31.02	1669	Brandt
Platinum.....	Pt	193.41	194.89	1750	Watson
Potassium.....	K	38.82	39.11	1807	Davy
Selenium.....	Se	78.42	79.02	1817	Berzelius
Silicon.....	Si	28.18	28.40	1822	Berzelius
Silver.....	Ag	107.11	107.92	Ancient	.....
Sodium....	Na	22.88	23.05	1807	Davy
Strontium	Sr	86.95	87 61	1808	Davy
Sulfur.....	S	31.83	32.07	Ancient	.....
Tin.....	Sn	118.15	119.05	Ancient	.....
Zinc.....	Zn	64.91	65.41	16th century	Paracelsus ?

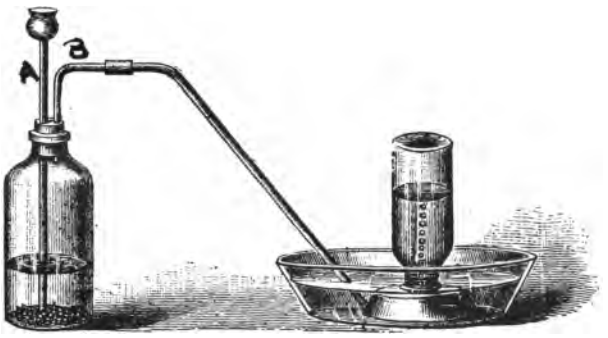
NONMETALLIC ELEMENTS.

HYDROGEN.

SYMBOL H. ATOMIC WEIGHT 1.

Name from Hydrogenium (water builder).

I. PREPARATION.--Make a gas generator, from a wide mouthed bottle, thus: Select a cork to fit the bottle snugly and bore two holes of a size to fit the funnel tube (A) and



the delivery tube (B). (Bend glass by heating in the broad gas flame used for illuminating). Take care that the funnel tube and delivery tube fit exactly.

Now put in the bottle enough granulated zinc (Zn) to cover the bottom, and through the funnel pour dilute sulfuric acid (H_2SO_4) until the action begins.

(To make a dilute solution pour one part of concentrated acid into four parts of water. *Always* pour sul-

furic acid into water, but *never* water into the acid, because so much heat is developed in the latter process that acid may be thrown out of the vessel by production of steam).

An evolution of gas at once begins and it may be collected, thus: Fill a bottle full of water and cover with a glass slip, then invert in a vessel containing water. On removing the glass slip the bottle remains full. Conduct the gas through the delivery tube into this bottle and the water will be displaced by the gas. In this way collect several bottles of gas and after covering with glass slips set them aside for future use. This process is called "collection by displacement of water."

II. Collect one or two bottles of the gas by placing the end of the delivery tube in an inverted bottle. Such a process is called "collection by upward displacement." What must be the weight of hydrogen as compared with air to make such a process possible?

III. Attach a clay pipe to the generator by means of a rubber tube. Make soap bubbles and set them free. What do they do? Is the gas in them heavier than air? Blow some bubbles from the lungs and set them free. Note the difference between these and those filled with hydrogen. Ignite some hydrogen bubbles when they are some distance away from the generator. Now collect a test tubeful of gas by displacement of water and ignite. Does the hydrogen explode? If it does there is still air mixed with the gas. Allow the gas to generate for a minute or two and test again. When the hydrogen is perfectly free from air perform experiment IV.

NOTE.—To make soap bubbles use a solution of soap made as follows. Cut about four ounces of pure

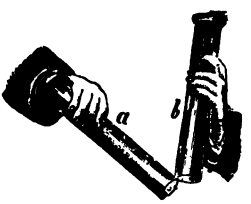
castile or palm oil soap into thin shavings and place them in a quart bottle. Fill with distilled water and shake until a saturated solution of soap is formed. Allow to stand for a short time and you should have a perfectly clear solution. If this is not the case shake up again with more water. Now to two volumes of soap solution add one volume of pure concentrated glycerin and a solution is obtained which makes splendid bubbles. Ivory soap has been used with much success in making this solution.

IV. Replace the delivery tube with a jet tube, i. e., a glass tube drawn out to a fine point. If you are sure that no air is present light the jet of gas. *Be very careful in this experiment so that dangerous explosions may be avoided.* A mixture of air and hydrogen is very explosive.

V. Hold a large tube or broken retort stem over the little burning jet, moving tube up and down. Note the singing noise. Try several tubes. Explain.

VI. Thrust a burning match into a bottle of hydrogen. Will the gas burn? Will it support combustion? Note color of the flame when first lighted, then after it has been burning some time. Is the flame hot?

Next invert a small bottle of gas under a burning match. Does the gas burn? What forces the gas out of the bottle?



VII. Hold an empty bottle inverted and invert another filled with hydrogen under it. Test the gas in each bottle. What change has taken place? What other experiments show the same properties of hydrogen?

VIII. To make a hydrogen gun. In the side of a baking powder can punch a hole near the bottom. Fill the can with hydrogen, over water, holding the thumb over

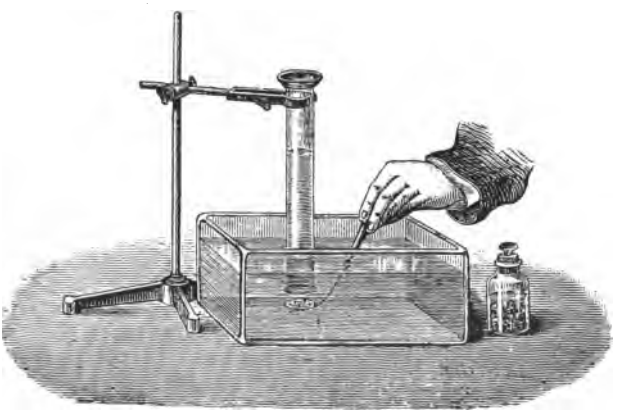
the vent while filling. When the can is full bubbles will come out around the edge. Put the cover on the can, hold the gun mouth downward, remove the thumb and apply a match to the vent. Wait till all the gas is burned. It may take some time, but you will know when the experiment is completed.

Water is formed every time that a mixture of air and hydrogen explodes, according to the equation $2H$ (hydrogen) $+ O$ (oxygen) $= H_2O$ (water).

IX. Repeat the above experiment, filling the gun with a mixture of half air and half hydrogen. Is there any difference in the way the two burn? Note caution under experiment IV.

FORMATION OF HYDROGEN FROM WATER.

X. Fill an evaporating dish or tin pan with water and invert in it a bottle or large test tube filled with water



as in experiment I. With a pair of dry forceps hold a small bright piece of metallic sodium (Na) under the mouth of the bottle after having first wrapped the sodium

in dry tissue paper. As soon as the evolution of gas stops test the contents of the bottle with a flame. What gas has been formed?

The reaction is as follows. H_2O (water) + Na (sodium) = H (hydrogen) + NaOH (sodium hydroxid).

Caution.—Sodium is a metal which combines very readily with water with evolution of hydrogen. The reaction is almost explosive, but if small bright pieces of the metal are used there is little danger. Before use, therefore, always see that the piece of metal is bright and clean. Sodium must be kept under benzine to prevent it from combining with the moisture of the air.

REACTIONS FOR FORMATION OF HYDROGEN.

Zn (zinc) + H_2SO_4 (sulfuric acid) = 2H (hydrogen) + $ZnSO_4$ (zinc sulfate).

Many other metals, such as iron (Fe) magnesium (Mg), etc., give hydrogen when dissolved in acids as sulfuric acid or hydrochloric acid (HCl.)

HALOGEN GROUP.

Cl (chlorin), Br (bromin), F (fluorin), and I (iodin). The word halogen means salt builder from the fact that the halogens combine with metals to form salts.

CHLORIN.

SYMBOL CL. ATOMIC WEIGHT 35.40.

Name from Chloros (green).

I. PREPARATION.—To a small quantity of manganese dioxid in a test tube add 5 or 6 cc. hydrochloric acid (HCl) and heat gently. Note the odor and color of the escaping gas. Reaction. MnO_2 (manganese dioxid) + 4 HCl = $MnCl_2$ (manganese chlorid) + 2 H_2O + 2 Cl (chlorin).

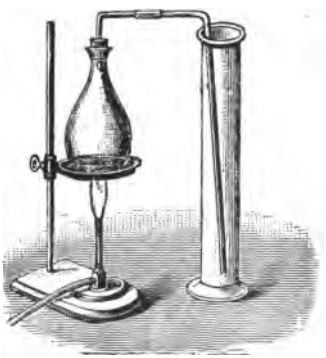
II. Hold a piece of moist blue litmus paper in the gas. Repeat with a moist piece of colored cloth or paper. Repeat same with dry pieces. What takes place in the first case? In the second? How does chlorin bleach?

NOTE.—Litmus is an organic coloring matter which is made blue by alkalis such as ammonia, potassium hydroxid, etc., and red by acids. If the litmus paper on your table should be red you can make it blue by moistening it, then holding it over the mouth of your ammonia bottle. Blue may be reddened by any acid.

III. Note carefully the odor of chlorin. Is the gas lighter than air? Try to pour some gas from one tube into another.

IV. Invert the tube of experiment I. in water. What happens? What do you now know about the solubility of chlorin in water?

V. Put a few crystals of potassium chlorate ($KClO_3$) in a test tube and add 5 or 6 cc. HCl. Heat gently and as soon as the gas begins to generate freely add 10 cc. of water. You have a solution of chlorin. Add a few drops to an indigo solution, to a litmus solution. What happens?



Reaction $4\ HCl + 2\ KClO_3$ (potassium chlorate) $= 2\ KCl$ (potassium chlorid) $+ 2\ H_2O + Cl_2O_4$ (chlorin tetroxid) $+ 2\ Cl$.

VI. Insert a lighted taper in a tube of the gas. Does chlorin burn or support combustion?

TO MAKE CHLORIN IN QUANTITY.

Mix well five parts manganese dioxid with eleven parts of common salt. Place in a flask fitted with delivery tube and treat with a dilute solution of sulfuric acid, one part water to two parts acid. Collect the gas by downward displacement, i. e., by conducting the gas into empty jars standing upright as in the figure. It cannot be collected over water as hydrogen was. Why?

Reaction. $MnO_2 + 3\,H_2SO_4 + 2\,NaCl$ (salt or sodium chlorid) $= 2\,NaHSO_4$ (sodium acid sulfate) $+ MnSO_4$ (manganese sulfate) $+ 2\,H_2O + 2\,Cl$.

BROMIN.

SYMBOL BR. ATOMIC WEIGHT = 79.34.

Name from Bromos (a stench).

I. PREPARATION.—Pulverize two or three crystals of potassium bromid and mix well with an equal quantity of manganese dioxid. Add about 5 cc. H_2SO_4 and heat gently. Dilute the acid by mixing one part of acid with five of water.

II. Note the color of gas, then very *carefully* its odor. The odor of bromin is, as indicated by the name, very unpleasant so be careful not to inhale too much. Test the gas with a burning splint. Test as you did chlorin with a piece of blue litmus paper and with colored cloth or paper. Is there any similarity in the action toward dyes between chlorin and bromin?

III. Dissolve a few crystals of potassium bromid (KBr) in water and add to the solution a few drops of chlorin water. Note change in color. Bromin has been formed according to the reaction $KBr + Cl = KCl$ (potassium chlorid) $+ Br$.

Now add a few drops of carbon disulfid (CS_2) to the solution and shake well. Put your thumb over the end of the test tube and shake up and down violently. Note change in color of the carbon disulfid.

Make a thin starch paste and add a little to a solution containing bromin. Note change in color after shaking. Compare with CS_2 test. These two tests are most frequently used for bromin in qualitative analysis.

IV. Invert the tube of experiment I. in water. Is bromin soluble?

REACTION FOR BROMIN.

$2\ KBr + MnO_2 + 3\ H_2SO_4 = 2\ KHSO_4$ (potassium acid sulfate) $+ MnSO_4 + 2\ H_2O + 2\ Br$. Compare with the equation for chlorin.

IODIN.

SYMBOL I. ATOMIC WEIGHT 125.89.

The name comes from a Greek word meaning violet colored.

I. PREPARATION.—Heat a few crystals of potassium iodid mixed with MnO_2 and H_2SO_4 just as you did in the preparation of bromin. Dilute the acid before use. Write the equation.

II. Test the gas with a burning splint. With blue litmus paper. Note the odor and color of iodin vapor. Is the vapor heavier or lighter than air? Is iodin ordinarily a solid, liquid or gas?

III. Invert the tube of experiment I. in water. When the tube is cool examine the sides for crystals of iodin.

What do you know about the relative solubility of Cl, Br and I.

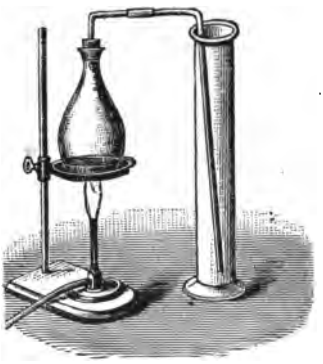
IV. Make a solution of potassium iodid in water. Add chlorin water and test with carbon disulfid and starch paste. Compare with bromin.

HYDROCHLORIC ACID.

FORMULA HCL. MOLECULAR WEIGHT 36.4.

The molecular weight of a compound is found by taking the sum of the atomic weights making up the compound. Cl (35.4) + H (1) = HCl (36.4).

I. PREPARATION.—Pulverize five grams of sodium chlorid and place in a flask arranged as in the figure, i. e., with a cork containing a single hole in which a delivery tube fits snugly. Pour about 10 cc. of concentrated H_2SO_4 on the salt and heat gently. Gas is generated and may be collected by downward displacement. Collect two bottles of the gas and cover with glass slips, then set aside.



NOTE.—In working with HCl it is absolutely necessary that the bottles in which the gas is collected be dry; otherwise gas is absorbed by the moisture clinging to the sides of the bottles and poor results are obtained.

REACTION FOR FORMATION OF HCl.

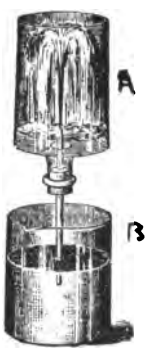
$NaCl + H_2SO_4 = NaHSO_4$ (sodium acid sulfate) + HCl, or if there is an excess of NaCl,

$2\ NaCl + H_2SO_4 = 2\ HCl + Na_2SO_4$ (normal or secondary sodium sulfate). *Note.*—An eight inch test tube may be used instead of the flask shown in the cut.

II. To make a jet tube, hold a piece of glass tube in the smoky flame of the gas, and as soon as soft remove

and draw out until about a mm. thick. Allow to cool, then scratch the narrow part with a file, break and you have two jet tubes. Select a cork to fit snugly a medium sized bottle or flask (A). Bore one hole and insert the jet tube small end in. Fill the dry flask with HCl gas, first removing the stopper. You can tell when the flask is full by holding over its mouth a rod dipped in ammonia (NH_4OH). White vapors will be formed when the acid comes in contact with the ammonia.

Insert the stopper once more then dip the end of the jet tube in a beaker filled with blue litmus solution. In a short time the solution will rush up into the bottle. What is the cause? Note change in color of the litmus. Why is it thus changed? Can HCl be collected over water?



III. Test a bottle of gas with moist litmus paper, with a burning splint. Note carefully the odor.

IV. Hold a rod dipped in ammonia over the mouth of a bottle of gas. What takes place.

Reaction $NH_4OH + HCl = H_2O +$ NH_4Cl (ammonium chlorid).

V. Hydrochloric acid as already indicated is very soluble in water. Make a solution by passing the gas into five cc. H_2O in a test tube for several minutes.

To a small portion of this solution add a drop of silver nitrate solution ($AgNO_3$). What happens?

Reaction. $AgNO_3 + HCl = \underset{\text{WHITE}}{\underline{AgCl}}$ (silver chlorid) + HNO_3 (nitric acid).

Try the same with a solution of lead acetate $Pb(C_2H_3O_2)_2$.

Reaction. $Pb(C_2H_3O_2)_2 + 2\ HCl = \underset{\text{WHITE}}{PbCl_2}$ (lead chlorid) $+ 2\ HC_2H_3O_2$ (acetic acid).

Repeat with mercurous nitrate.

Reaction. $HgNO_3 + HCl = \underset{\text{WHITE}}{HgCl}$(mercurous chlorid) $+ HNO_3$.

NOTE.—In all these experiments a single drop will suffice. Never use large quantities of reagents unless so directed.

VI. Repeat experiment V. with a solution of NaCl in water. Write out all the reactions.

VII. Solubility of metals in hydrochloric acid.

Dissolve a little piece of iron wire in the acid from your table. Is a gas given off? Test with a burning splint. What is it? Repeat with Zn.

In a small evaporating dish evaporate these solutions to dryness. Note what you have left from each solution after evaporation. You have two salts, one of iron and one of zinc. Hydrochloric acid dissolves metals to form salts. Not all metals are soluble in HCl, though many are.

VIII. Hold a green leaf in HCl gas.

IX. Invert the tube or flask of experiment I. in water. Which is more soluble, the halogen family or this compound of a halogen with hydrogen?

X. Put some gold leaf in a test tube and add HCl from the table. Note effect.

XI. Put another piece in a second tube and treat with nitric acid (HNO_3). Note effect.

Mix the two and note change.

A mixture of nitric and hydrochloric acids is called Aqua Regia. Compare with reaction for dissolving mercury in Aqua Regia.

HYDRIODIC ACID.

I. PREPARATION.—Put two or three crystals of iodin in a test tube, then add water till it is half full. From a Kipp's apparatus run in hydrogen sulfid (H_2S) until the solution becomes colorless. Boil to free from excess of H_2S, i. e., boil till the liquid no longer smells of the gas. You now have a weak solution of hydriodic acid containing sulfur in the form of a fine powder. After standing a moment the sulfur will settle and the clear solution may be used for the following experiment.

Reaction $2\ I + H_2S = 2\ HI + S$ (sulfur).

For description of Kipp's apparatus see hydrogen sulfid.

II. Add a few drops of the hydriodic acid solution to a solution of mercuric chlorid ($HgCl_2$). Note the color of the precipitate.

Reaction $HgCl_2 + 2\ HI = \underbrace{HgI_2}_{\text{CRIMSON}}$ (mercuric iodid) $+ 2\ HCl$.

Try same with a solution of silver nitrate, of lead acetate.

Reactions $AgNO_3 + HI = \underbrace{AgI}_{\text{PALE YELLOW}}$ (silver iodid) $+ HNO_3$.

$Pb\ (C_2H_3O_2)_2 + 2\ HI = \underbrace{PbI_2}_{\text{YELLOW}}$ (lead iodid) $+ 2\ HC_2H_3O_2$.

III. Try II. with a solution of potassium iodid and write out all the reactions.

HYDROFLUORIC ACID.

Hydrofluoric acid is an extremely volatile and poisonous acid. In working with it be very cautious, always work under a hood and never inhale any of the fumes. A hood is a cupboard connected with a chimney, so that all noxious vapors are drawn off into the air above the building. It is well always to test a hood before use by light-

ing a match, holding inside the hood and noticing by the flame and smoke if the draught is good.

I. PREPARATION.—Etching experiment.

Cover a glass slip on one side with wax or paraffin. Cut in the wax with a pin or some sharp pointed instrument the design that you wish engraved. In a leaden dish under the hood place about four grams of powdered calcium fluorid (CaF_2) and add strong sulfuric acid so as to make a thin paste. Warm very gently and cover the dish with the piece of glass, wax side down. After standing about half an hour the design you have drawn will be etched on the glass. Scrape off the wax and clean glass thoroughly.

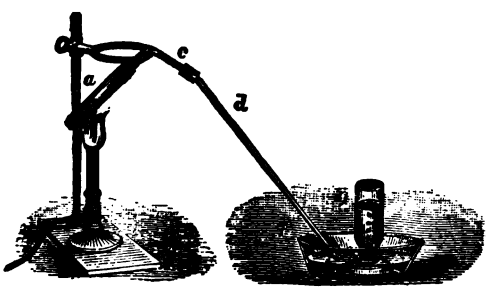
Reaction. $CaF_2 + H_2SO_4 = 2\ HF$ (hydrofluoric acid) $+ CaSO_4$ (calcium sulfate).

Compare with the reaction for hydrochloric acid.

OXYGEN.

SYMBOL O. ATOMIC WEIGHT 16.

I. PREPARATION.—Pulverize one-quarter of a test tubefull of potassium chlorate. Add half as much man-



ganese dioxid and mix thoroughly. Prepare a large test tube with a tight fitting cork and delivery tube as shown in the figure. Place the mixture in the tube and arrange to collect the gas over water.

II. Heat the mixture gently, being careful not to melt the tube by heating too strongly in one place. It is always well to heat a tube in the smoky flame of the Bunsen burner before heating strongly. This deposits a coating of soot or carbon on the tube which prevents breaking. Collect three or four small bottles of the gas and set aside after covering with glass slips, then collect two large bottles of gas.

III. Into a small bottle thrust a glowing splint. If it relights, extinguish and relight as often as possible. Will oxygen burn? Will it support combustion?

IV. Make a deflagrating spoon by hollowing out a piece of crayon or electric light carbon with a knife. Make a handle by wrapping a piece of wire about the chalk. In the deflagrating spoon place a little piece of sulfur and ignite in the Bunsen burner and thrust in a bottle of oxygen as in the figure.

Note the color of the flame and the odor of product formed.

Reaction sulfur S + 2 O = SO_2 (sulfur dioxid).

V. Place a glowing piece of charcoal in the spoon and lower it into a bottle of oxygen. Carbon dioxid is formed according to the equation C + 2 O = CO_2 carbon dioxid).

VI. Many substances which are incombustible under ordinary conditions burn easily in oxygen. Coil a fine iron wire in the form of a spiral. Warm one end slightly and dip in sulfur. Ignite and thrust in a bottle of oxygen. The iron takes fire and burns brilliantly. It is always necessary to have about an inch of water in the bottom of the bottle. Why?

Reaction. $3\ Fe\ (iron) + 4\ O = Fe_3O_4$ (ferric oxid).

VII. Dry a piece of phosphorus carefully with blotting paper and place in the deflagratiug spoon. Lower into a bottle of oxygen and ignite with a warm wire.

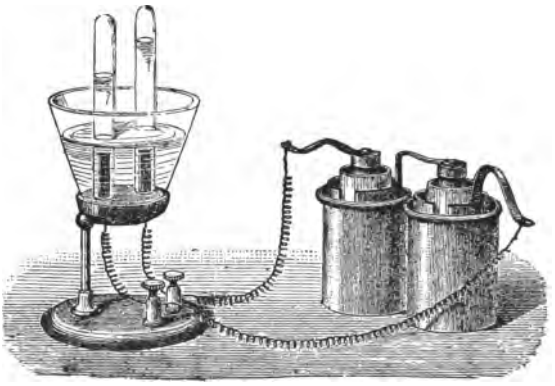
In handling phosphorus extreme care must be used, as it takes fire very easily. *Never* handle it with your fingers but *always* with pincers.

WATER.

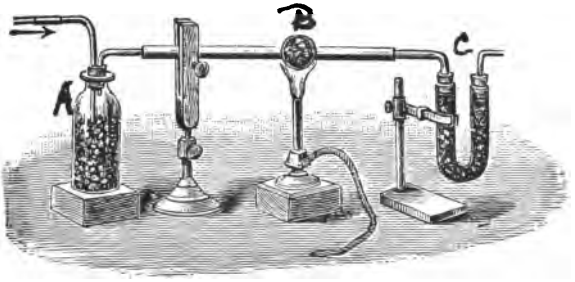
COMPOSITION OF WATER PROVED BY EXPERIMENT.

I. Crack off the bottom of a medium sized, wide-mouthed bottle or glass jar. This may be done by scratching the glass with a file, then touching the crack with a red hot glass rod or piece of iron. The bottle will break parallel to the first file mark. Close the neck with a close fitting cork, through which two platinum wires pass. Put tips on these wires of small pieces of platinum foil about one-half inch in length by one-quarter wide. Invert the apparatus thus made, support it in some way and fill two-thirds full of water; add two or three cc. of H_2SO_4 and connect the outer ends of the platinum wires with two potassium dichromate or other good cells. Over the platinum tips invert two graduated tubes filled with water. After connecting with the cells bubbles of gas will form on the platinum tips and rise in the tubes. After several cubic centimeters of gas have collected note the

ratio between the amounts of gas in the two tubes. Test each tube with a flame. What are the gases? Of which gas is there the greater quantity? Which gas came off at the + pole of the battery?



II. Pass hydrogen over heated copper oxid (CuO) in a glass tube. $CuO + 2\ H = H_2O + Cu$ (copper). Water will be formed according to the equation given. In the given figure B is the glass tube in which a bulb has been



blown to contain the copper oxide. A is a bottle filled with pieces of pumice stone which have been soaked in concentrated sulfuric acid. This removes all moisture from the hydrogen. C is a tube filled with calcium

chloride ($CaCl_2$) which absorbs all moisture passing over from the bulb. The amount of water formed by treating a certain amount of copper oxid with hydrogen may be determined by weighing the calcium chlorid tube before and after the experiment.

SULFUR.

SYMBOL S. ATOMIC WEIGHT 31.98.

I. Note the remarkable changes in sulfur under the action of heat. Fill a six inch test tube half full of sulfur and heat in the flame. At about 110° it forms a light yellow liquid. It becomes thick at 150° and the color changes to almost a black. At a still higher temperature, 330°—340°, it becomes more fluid but still retains its dark color. At a still higher heat it is changed into a red colored vapor. The boiling point of sulfur is about 448° Centigrade.

When the experiment is finished pour the sulfur remaining in the tube into a beaker or evaporating dish containing cold water. An amorphous variety is formed which is much like rubber.

II. Using the same test tube as for the above experiment make sulfid of iron, thus: Mix well two grams of flowers of sulfur (fine sulfur) with three grams of fine iron filings, place in a test tube and heat the lower end of the tube. When the mixture begins to glow remove from the flame and the ignition will go on of itself. Note what

remains in the tube after heating. Does it look like sulfur? Like iron filings? Break the tube and take out a little piece of the sulfid you have made and treat it with dilute hydrochloric acid in a small test tube. Note the odor of gas generated. What does it smell like? The gas is hydrogen sulfid.

Reaction. $Fe + S = FeS$ (ferrous sulfid).

III. Burn a very small quantity of sulfur in the air on a deflagrating spoon. Note the color of flame and odor of gas formed. What does the gas smell like? It is called sulfur dioxid and is made by a direct union of sulfur and the oxygen of the air.

Reaction. $S + 2\,O = SO_2$.

IV. Light a quantity of sulfur in a deflagrating spoon and lower into a large bottle. When the sulfur stops burning what does the bottle contain? Lower a moist colored flower into the bottle and note change in color. Do you know of any way in which this property of sulfur dioxid is put to practical use?

V. Prepare dilute sulfuric acid by putting about three drops of acid in three cc. of water. Place the bleached flower in this solution. Does the color change again?

VI. Suspend a moist wheat straw in a bottle of SO_2 as in IV.

Sulfur dioxid takes up oxygen from water and gradually changes to sulfuric acid. At the same time it sets free hydrogen and this nascent hydrogen by its action upon the coloring matter either destroys or changes the color. Such a process is called reduction while an action thguorb about by oxygen is called oxidation.

VII. Sulfur is probably formed in nature as in the following experiment: H_2S (hydrogen sulfid) and SO_2 (sulfur dioxid) are two gases always found in volcanic regions. When they are brought together they form free sulfur and water according to the equation.

$$2\ H_2S + SO_2 = 2\ H_2O + 3\ S.$$

Conduct hydrogen sulfid from a Kipp's apparatus (see description under hydrogen sulfid) into an empty bottle and at the same time sulfur dioxid from a generator (see sulfuric acid manufacture). A deposit of sulfur is formed on the bottom and sides of the bottle.

HYDROGEN SULFID.

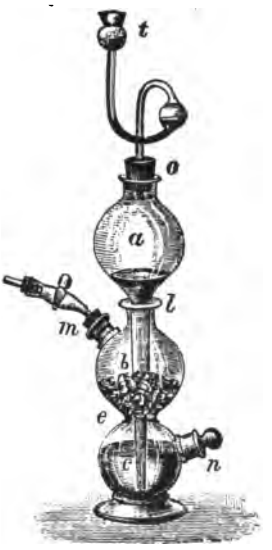
I. PREPARATION.—Fit a six inch test tube with a cork through which passes a jet tube with the small end out. In the test tube place a small piece of ferrous sulfid (Fe S) and fill half full of water. Add several drops of sulfuric acid and soon an evolution of gas will begin. Light the gas at the end of the delivery tube. What is formed? Compare odor of gas formed by burning with that of the original gas. Test the hydrogen sulfid with moist blue litmus. From this test how would you class H_2S among the compounds you have already studied?

Equation. $FeS + H_2SO_4 = FeSO_4$ (ferrous sulfate) $+ H_2S$.

Hydrochloric acid may be used as well as sulfuric, so try to write the equation.

This same method is used to prepare the gas in quantity, only a more complex piece of apparatus is used. The figure represents the apparatus used, which is designated as Kipp's apparatus from its inventor. It consists of three large bulbs of glass, the two lower bulbs (b) and (c) being joined together while (a) having a long stem like

a funnel is made to fit into (b) snugly and extends nearly to the bottom of (c) the lower bulb. The apparatus to be used must be charged as follows: First introduce a piece of glass wool or loose asbestos through the opening at (m), then through the same opening fill bulb (b) half full of large pieces of ferrous sulfid. The glass wool prevents small pieces of sulfid from falling into the lower bulb (c). Next close the opening (m) with a cork carrying a glass tube with a stop cock. In the top of the first bulb (a) place a cork containing a safety tube (t). Now through this tube pour a mixture of one part sulfuric acid to eight or ten of water, or hydrochloric acid (one to three) until the lower bulb is full and the acid covers a portion of the ferrous sulfid. Hydrogen sulfid is generated and escapes through the tube at (m). When you wish to stop the apparatus close this stop cock and the pressure of the gas forces the acid down into the bulb (b) and from there into bulb (a). The opening at (n) is intended for use in cleaning out the apparatus.



Such an apparatus should be used under a hood as the hydrogen sulfid is very poisonous and should never be inhaled.

II. Pass gas from a Kipp's apparatus into test tubes containing solutions of (a) copper sulfate ($CuSO_4$), (b) mercuric chlorid ($HgCl_2$), (c) lead acetate ($Pb(C_2H_3O_2)_2$), (d) arsenic oxide (As_2O_3). Note colors in each case.

Reactions. $CuSO_4 + H_2S = CuS$ (copper sulfid) $+ H_2SO_4$.

$HgCl_2 + H_2S = HgS$ (mercuric sulfid) $+ 2\ HCl$.

$Pb(C_2H_3O_2)_2 + H_2S = PbS$ (lead sulfid) $+ 2\ HC_2H_3O_2$.

$As_2O_3 + 3\ H_2S = As_2S_3$ (arsenic sulfid) $+ 3\ H_2O$.

III. Pass hydrogen sulfid into a solution of ferric chlorid ($FeCl_3$). It is reduced to ferrous chlorid and sulfur is precipitated.

Reaction. $2\ FeCl_3 + H_2S = 2\ HCl + 2\ FeCl_2$ (ferrous chlorid) $+ S$.

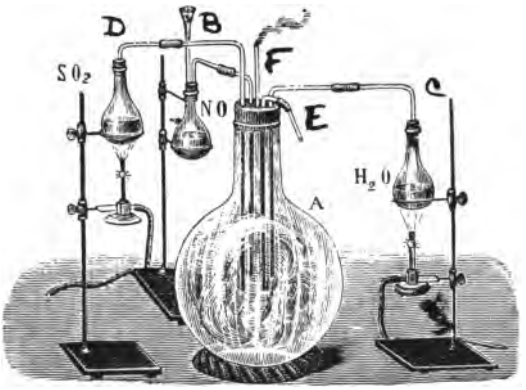
IV. Pass hydrogen sulfid through strong sulfuric acid. What is formed? Collect the precipitate if one is formed and ignite on a small piece of wood. Note the odor and color of flame of the burning precipitate. What was the precipitate? Try the same with a mixture of nitric and hydrochloric acids.

V. Give all possible means at your disposal of detecting (a) SO_2, (b) H_2S and (c) O.

SULFURIC ACID.

I. Preparation.—Sulfuric acid may be made on a small scale as follows. A large glass globe A is closed with a cork containing five glass tubes as shown in the figure. Four of these tubes reach nearly to the bottom of the globe, while one serves as an escape valve F. One of the tubes is connected with a flask D containing strong sulfuric acid and copper turnings for the generation of sulfur dioxid. Another, B, contains copper turnings and dilute nitric acid 1–3 for the generation of nitric oxid and the last C contains water for the formation of steam. The tube marked E is used to renew the supply of air from time to time during the process.

Begin the experiment by heating flask D. When this reaction begins pour dilute nitric acid into B (one part acid to three of water) and allow steam to enter the large bulb from C. It is well to have the water in C almost boiling when the nitric acid is placed in B. The main thing is



to so time the reaction as to allow neither sulfur dioxid nor nitric oxid to escape into the room. The reactions which take place in the various parts of the apparatus are as follows.

D. $Cu + 2\ H_2SO_4 = CuSO_4$ (copper sulfate) $+ SO_2 + 2\ H_2O$.

B. $3\ Cu + 8\ HNO_3$ (nitric acid) $= 3\ Cu\ (NO_3)_2$ (copper nitrate) $+ 2\ NO$ (nitric oxid) $+ 4\ H_2O$.

A. $NO + O$ (oxygen from the air) $= NO_2$ (nitrogen dioxid sometimes called tetroxid).

A. $SO_2 + NO_2 = SO_3$ (sulfur trioxid) $+ NO$.

A. $SO_3 + H_2O = H_2SO_4$ (sulfuric acid).

The reaction is claimed by many chemists to be much more complex than here indicated, but the above is a general explanation of what takes place in any sulfuric acid factory. The supply of oxygen must be renewed in the

globe A whenever the red fumes of nitrogen dioxid can no longer be seen.

II. After some acid has collected in the bottom of the receiver allow the apparatus to cool and test a small quantity with barium chlorid ($BaCl_2$). Note the precipitate. Try the same with dilute acid from your table. Are the two results the same?

Reaction. $BaCl_2 + H_2SO_4 = BaSO_4$ (barium sulfate) $+ 2\ HCl$.

III. To a small quantity of sugar in a test tube add concentrated sulfuric acid. Note color of sugar. Try the same with a match stub.

IV. To a small quantity of water in a test tube add sulfuric acid. Note change in temperature of the water. Sulfuric acid has a great affinity for water, and it is on this that the blackening of the sugar and the wood depends. These substances are organic bodies composed of oxygen, hydrogen and carbon. The acid takes away some of the oxygen and hydrogen and leaves the carbon.

NITROGEN.

SYMBOL N. ATOMIC WEIGHT 14.

I. PREPARATION.—In a slice of cork about half an inch thick and smaller than the mouth of a large bottle or jar, make a cavity and line with plaster of Paris. Make a paste by mixing the plaster with a little water. When the plaster is dry, place a dry piece of phosphorus about as large as a pea on the cork, float it on water in a basin filled with water, ignite the phosphorus with a hot wire and invert the bottle over it.

Reaction. $2\ P$ (phosphorus) $+ 5\ O = P_2O_5$ (phosphorus pentoxid).

$P_2O_5 + 3\ H_2O = 2\ H_3PO_4$ (phosphoric acid).

II. Note carefully the burning of the phosphorus. At last it will stop burning, although it may not all be consumed. Why? What is necessary in the air for combustion?

Note the white vapor. It is phosphorus pentoxid. It will be gradually absorbed and the gas in the bottle will become transparent, then you have nearly pure nitrogen left.

III. Remove the bottle from the water, using a glass slip to cover it. Insert a burning splint. Does nitrogen burn or support combustion? What caused the phosphorus to burn? Why do we need any nitrogen in the air?

IV. Allow the bottle to stand for five or ten minutes, then test with the flame once more. Is the nitrogen as pure as at first?

V. Test the water from experiment I–II with blue litmus. Taste of a drop. What is it? See the equations under I.

AMMONIA.

I. PREPARATION.—Pulverize about a gram of ammonium chlorid (NH_4Cl) and note its odor.

II. Place a small quantity of lime ($Ca(OH)_2$) in your hand and note its odor. Mix the chlorid and lime and note odor again. Is there any difference?

III. Remove from the hand to a dry test tube and heat high above the Bunsen flame, having first smoked the tube in the smoky flame.

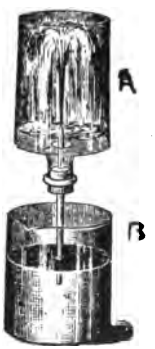
Reaction. $2\ NH_4Cl$ (ammonium chlorid) $+ Ca(OH)_2$ (slaked lime) $= CaCl_2$ (calcium chlorid) $+ 2\ NH_3$ (ammonia) $+ 2\ H_2O$.

This reaction works as well with unslacked lime (CaO). In the latter case a less amount of water is formed. Write the equation. This reaction is a special case of a very general reaction. Nearly any ammonium salt heated with a fixed alkali such as potassium hydroxid (KOH) or sodium hydroxid (NaOH) will set free ammonia.

IV. Bring a glass rod moistened with HCl to the mouth of the test tube generating ammonia. What is formed? Write the equation.

V. Insert a piece of moist red litmus paper in the tube. How does this reaction differ from a similar one under hydrochloric acid?

VI. Pulverize about two grams of NH_4Cl in a morter and mix well with about twice as much lime. Place the mixture in a large test tube to which a delivery tube has been attached.



VII. Prepare a bottle with a jet tube as in one experiment under hydrochloric acid and perform the same experiment, using ammonia instead of HCl to fill the bottle and a red litmus solution instead of a blue. Is ammonia very soluble in water?. What is the ordinary ammonia of commerce?

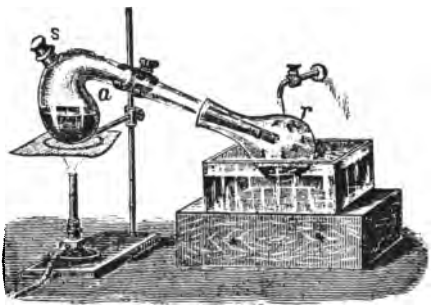
VIII. Insert a burning splint in a bottle or test tube containing ammonia. Does the gas burn or support combustion?

NITRIC ACID.

I. PREPARATION.—In a glass retort of about 250 cc. capacity put a small test tubeful of pulverized potassium nitrate (KNO_3), or, sodium nitrate ($NaNO_3$).

Through the top of the retort pour twenty-five cc. of strong H_2SO_4 on the nitrate. Place the retort on a piece of asbestos paper on a ring stand, clamp securely and heat gently.

Condense the vapors of nitric acid by allowing them to pass into a flask or small bottle placed in a pan of water. Change the water from time to time, or apply a wet sponge to the bottle to keep it cool. The end of the retort neck should be thrust well into the receiver as shown in the figure.



Be careful not to heat too much or the liquid in the retort will froth violently and may spoil the experiment.

Stop the reaction when the amount of acid that distills over becomes small.

Reactions. $NaNO_3$ (sodium nitrate) + H_2SO_4 = $NaHSO_4$ (sodium acid sulfate) + HNO_3.

If much sodium nitrate is present and the temperature is high a second reaction takes place, thus:

$NaHSO_4 + NaNO_3 = Na_2SO_4 + HNO_3$ and normal sodium sulfate is formed.

II. Pulverize a little charcoal, heat in an iron spoon and when hot pour a little of the nitric acid you have made on it.

The charcoal burns with bright sparks.

NOTE.—Nitric acid is called a strong oxidizing agent because it supplies oxygen to support combustion.

III. Dip a piece of woolen cloth into dilute nitric acid from the table. After standing a short time wash the cloth thoroughly. Note its color.

IV. Place a piece of copper foil (Dutch leaf) in a test tube and pour on a little nitric acid.

V. Cover a piece of iron or brass with paraffin. Write upon it with the point of a file or pin; pour a little nitric acid upon the writing and let it stand until bubbles are formed along the lines of writing. Wash, remove the paraffin. Note the result.

VI. Place a cubic centimeter of indigo solution in a test tube and pour on nitric acid. This is often used as a qualitative test for nitric acid.

VII. Into dilute nitric acid drop a crystal of ferrous sulfate ($FeSO_4$). Do not shake. Note color around the crystal. Try the same with five cc. of H_2O and one cc. of HNO_3.

VIII. Brown ring test for nitric acid and nitrates.

Dissolve a little KNO_3 or $NaNO_3$ in water. Add a little ferrous sulfate solution. Incline the test tube and allow a little strong H_2SO_4 to run down the side. Note effect where the acid and other liquids meet. This is the most common test for nitric acid and the nitrates.

Reaction. The brown ring test may be represented in an equation thus: $2\ KNO_3 + 4\ H_2SO_4 + 10\ FeSO_4 = K_2SO_4$ (potassium sulfate) $+ 3\ Fe_2(SO_4)_3$ (ferric sulfate) $+ 4\ H_2O + 2\ (FeSO_4)_2NO$.

The last compound has no specific name and its composition is involved in uncertainty. (Prescott & Johnson).

IX. Put one drop of HNO_3 in a test tube of water and taste the mixture from a glass rod. Test the same with blue litmus paper.

NITROUS OXID.

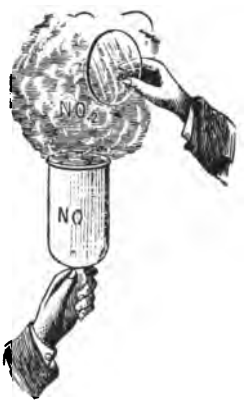
I. PREPARATION.—In a large test tube place half a four inch test tubeful of ammonium nitrate (NH_4NO_3) and arrange a delivery tube. Heat gently and collect three bottles of the gas, two over warm water and the other by downward displacement. In heating two things *must* be observed. First. Do not heat too hot, as dangerous explosions may occur. Heat so that a gentle evolution of the gas takes place. Second. Keep the test tube turned away from your face, so that the contents may not strike you should an explosion occur.

II. Note the odor of the gas and test one of the bottles collected over water and the one collected by downward displacement with a glowing splint.

III. Burn a piece of phosphorus in the remaining bottle of N_2O. Does it burn as well as in oxygen?

NITRIC OXID.

I. PREPARATION.—In a test tube place a few pieces of copper (Cu). Mix two cc. of HNO_3 and two cc. of H_2O and pour on the copper.



II. Collect the gas over water and test a bottle with a glowing splint.

III. Note the effect when air is admitted to a bottle of NO.

IV. Place the solution of copper in an evaporating dish and evaporate to dryness on asbestos paper. You have a salt. What salt?

Caution. The fumes of NO are very disagreeable, therefore, make small quantities and avoid letting much out in the room.

The reddish fumes formed when NO comes in contact with the air are fumes of nitrogen dioxid (NO_2).

PHOSPHORUS.

SYMBOL P. ATOMIC WEIGHT 31.

The name phosphorus comes from the Greek, meaning light bearer and has reference to the property of phosphorescence.

Before beginning this experiment read carefully the caution given on page 29. A phosphorus burn makes a disagreeable sore, very difficult to heal.

I. Rub a *small* piece of phosphorus between two pieces of board. The heat of friction will ignite the phosphorus.

II. Place a piece of phosphorus about as large as an apple seed in a six-inch test tube and fill one-half full of water. Drop in three or four large crystals of potassium chlorate ($KClO_3$). Insert a thistle tube, placing the lower end near or on the phosphorus, then pour two or three cc. of H_2SO_4 through the tube. Note the effect.

III. Touch a piece of phosphorus with a test tube filled with hot water.

IV. Dip a piece of paper in a solution of phosphorus in carbon disulfid (CS_2). Lay it on an iron ring or pan and watch it closely till chemical change occurs.

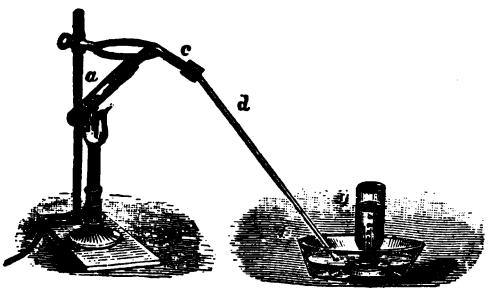
V. Heat a small quantity of lamp-black in an iron spoon and allow to cool. On a piece of paper or board lay a clean dry piece of phosphorus. Shake a quantity of lamp-black upon it and note the effect. Why does phosphorus smoke when taken out of water?

VI. Place a crystal of iodin on a dry piece of phosphorus, phosphorus iodid is formed (PI_3).

CARBON.

SYMBOL C. ATOMIC WEIGHT 12.

I. Arrange a six-inch test tube, an old one if you have it, with a delivery tube. Fill the tube with shavings or small pieces of wood, and heat. Collect the gas evolved over water and test with a lighted splint. When the gas



ceases to come off, close the tube with a cork, or plaster of Paris, and allow to cool. When cool examine the contents of the tube. What have you? Compare with charcoal. Heat a small piece of the charred wood in an iron spoon. Does it burn with flame? What causes the flame of burning wood?

II. Press down upon the luminous flame of a Bunsen burner a cold iron spoon. Note deposit on the bottom. Why is unburned carbon deposited on the spoon?

III. Make a mixture of one part powdered charcoal or lamp-black to eight of copper oxid (CuO) and heat strongly in a test tube. After heating some time, break the test tube and note the change that has occurred. The CuO has been reduced to metallic copper. Why?

IV. In each of four bottles of about 100 cc. capacity put about a tablespoonful of lamp-black. Into the first pour ten or fifteen cc. of a solution of iodin and starch. Into the second pour methyl orange or blue litmus or any vegetable coloring matter. Into the third pour hydrogen sulfid. Into the fourth a solution of potassium dichromate ($K_2Cr_2O_7$) or potassium chromate (K_2CrO_4).

Close each bottle with a cork, shake well and filter. If the filtrate is still colored repeat the operation until the solution is clear. The odor of the H_2S will be removed and the colors of all the solutions except one. That one is a mineral coloring matter. Which is it? Why is the color not removed? How does charcoal remove colors?

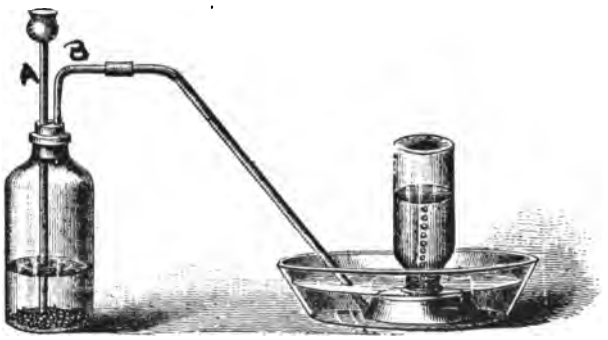
NOTE.—Save the mineral solution. It may be poured back into the original bottle and used over and over again.

CARBON DIOXID.

I. PREPARATION.—Arrange a generator in the same way as for hydrogen. Place in the bottle several small pieces of marble ($CaCO_3$). Cover these with water, and through the thistle tube pour small quantities of concentrated HCl. The gas may be collected over water or by downward displacement. The latter method is usually adopted. Collect several bottles full. In a bottle of the CO_2 thrust a piece of cotton soaked in alcohol and ignited. Does the CO_2 burn or support combustion? Could you live in an atmosphere of CO_2?

II. Pass a stream of CO_2 through a little lime water in a beaker. Notice color of precipitate. Continue until the color disappears.

III. Place a piece of candle at the lower edge of a sloping board and pour CO_2 on the board so as to run



down on the candle. What two properties of CO_2 does this experiment show?

IV. Pour a bottle of CO_2 upon a candle flame just as you might pour a glass of water on it.

V. Breathe through a solution of lime water until a precipitate is formed, and continue until it disappears. Find out from your text book why this precipitate disappears when the amount of CO_2 is in excess. Boil the solution after the milky color disappears and note result. Why is lime deposited in a tea kettle?

VI. In a bottle of air place a glowing piece of charcoal suspended on a wire. When the coal ceases to glow test the contents of the bottle for CO_2 by means of lime

water, i. e., pour in about two cc. of lime water and shake.

VII. Test the air of the room by filling a large bottle with it and shaking up with lime water. To get the air from the room carefully suck out air already in the bottle with a glass tube or fill by using an aspirator. In sucking out the air do not breathe in the bottle. Also test the air of the room by allowing a watch glass full of lime water to stand in the room for an hour or more. Note the surface of lime water. What has formed on it?

Reactions. $CaCO_3 + 2\ HCl = CaCl_2 + H_2O + CO_2$.

$CO_2 + Ca\ (HO)_2$ (calcium hydroxid or lime water) $= CaCO_3 + H_2O$.

$CaCO_3 + CO_2 + H_2O = CaH_2(CO_3)_2$ (calcium acid carbonate).

ETHYLENE OR ETHENE.

Carbon forms a vast number of compounds with hydrogen, but only a few of them can be experimented upon in an elementary course.

I. PREPARATION.—In a test tube arranged as for the preparation of nitrous oxid place a mixture of two cc. of alcohol (C_2H_6O) and eight cc. of strong sulfuric acid.

Extreme care must be used in mixing acid and alcohol because much heat is developed.

Pour the acid slowly into the alcohol and stir vigorously in the meantime.

Heat the mixture gently and collect the gas, C_2H_4, over water.

The mass in the test-tube becomes black and froths, therefore it must be watched carefully to prevent frothing over.

II. Test the C_2H_4 in the bottle to see whether it burns or supports combustion.

Force the gas out by pouring in water as it burns.

III. Make a jet-tube and light the gas as in the case of hydrogen.

What is formed when ethylene burns? Write the equation.

IV. Put a few drops of bromin water in a bottle of gas (C_2H_4). Shake well and note the liquid formed at the bottom of the bottle. It is ethylene bromid ($C_2H_4Br_2$). Note the odor and oily form of the liquid. The name olefiant gas is said to come from its property of forming oils with other elements.

Reactions. $C_2H_6O - H_2O = C_2H_4$.

Sulfuric acid has a strong affinity for water and takes away water from the alcohol; this leaves the gaseous compound C_2H_4. Other reactions take place so that the ethylene may have mixed with it CO_2 or SO_2 as well as ether and other compounds.

SUGAR.

Qualitative tests for cane sugar and glucose.

I. To a solution of cane sugar ($C_{12}H_{22}O_{11}$) add a little potassium hydroxid (KOH), then add a few drops of a dilute solution of cupric sulfate ($CuSO_4$) drop by drop. Warm very gently. Note the change in color, if any. It is important that the mixture should not be heated too much or too long, because the cane sugar may thus be

changed into glucose and the experiment spoiled. Use a four-inch test tube for this experiment.

II. Perform the same experiment with a glucose ($C_6H_{12}O_6$) solution. Note that the color is orange, or reddish brown.

III. Test an unknown solution in the same way.

IV. Test mixed candies and samples of sugar obtained from different stores.

V. To make sugar from starch.

1. Put two cc. of H_2SO_4 into 100 cc. of H_2O in a large evaporating dish of about three-hundred cc. capacity. Allow the mixture to cool.

2. Prepare twenty-five cc. of starch paste by adding starch ($C_6H_{10}O_5$) to twenty-five cc. of water until a thin paste is formed. Warm gently for about two minutes and add water if necessary to make the paste thin enough to flow readily.

3. Bring the solution of acid to boiling and add the starch solution drop by drop stirring continually till all is added. Boil slowly twenty or thirty minutes and then set aside to cool.

During the boiling add water if necessary.

4. Now add pulverized chalk or marble until effervescence ceases, to neutralize the H_2SO_4. Dip the end of a glass rod into the mixture and touch to the tongue to ascertain if the sour taste of the acid has disappeared.

5. The liquid has probably been changed to a pasty mass by the addition of chalk. Now thin with water and filter.

6. Evaporate the filtrate to dryness and you have a pasty mass of sugar.

Is it glucose or cane sugar?

Reactions. The $CuSO_4$ is reduced to Cu_2O, a reddish brown cuprous oxid. The acid is neutralized according to the reaction:

$$H_2SO_4 + CaCO_3 = CaSO_4 + CO_2 + H_2O.$$

The calcium sulfate is insoluble in water and is separated from the sugar solution by filtration.

VI. To make sugar out of sawdust or rags.

To two parts by weight of wood or clean linen rags add three parts of conc. H_2SO_4. The wood must be in the form of thin shavings or sawdust and the rags cut or torn into shreds. Add the wood or rags gradually to the acid and set aside for twenty-four hours. Dilute with twenty or thirty volumes of water and boil two or three hours, adding water as needed. Neutralize the acid with chalk, dilute with water, filter off the calcium sulfate, evaporate nearly to dryness and test.

What kind of sugar have you this time?

BORON.

SYMBOL B. ATOMIC WEIGHT 10.86.

I. Preparation of boric or boracic acid (H_3BO_3).

Dissolve half a test tubefull of borax in ten cc. of boiling water in a beaker or small evaporating dish and add to the solution 2.5 cc. of concentrated hydrochloric acid. As soon as the solution cools crystals of boric acid will be deposited on the bottom of the dish.

II. Test for boric acid.

Dissolve a little of the boric acid you have made in alcohol in an iron spoon. Ignite the alcohol and stir the burning solution with a glass rod. The flame will be colored green.

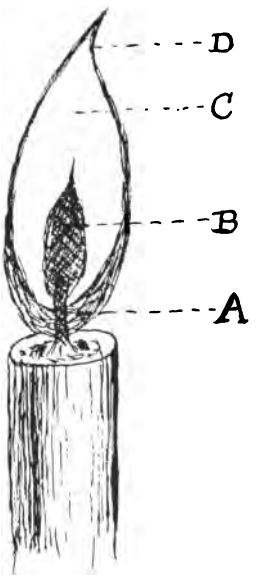
III. To make boric anhydrid (B_2O_3). In a clean iron spoon, heat some crystals of boric acid. As soon as

all the water has been driven off the anhydrid will be left as a clear liquid, which may be drawn out in long threads by touching to the surface of the liquid a stick or glass rod. When cold the anhydrid will resemble glass.

FLAME.

A. STRUCTURE.

I. Examine a candle flame for the four parts. A. The small blue cup-shaped portion of the flame at the base of the wick. This may be considered as merely a thick portion of the almost invisible blue mantle D. B. The more luminous cone in the very center of the whole flame. C. The luminous cone outside the central cone. D. The nearly invisible coat that surmounts the entire flame. This last is very hard to see. Draw a very accurate diagram of the candle flame showing all the parts. The heat of combustion of the first zone A converts into gas a large quantity of the fatty materials of the candle which are being drawn up by the wick. These gases form the second zone B. This zone is completely shut off from the air and therefore there is no combustion in it. You may prove this by putting a match head in this portion of the flame. The match will not ignite if the experiment is well performed. Make several trials. In the next portion of the flame C, the luminous cone, the supply of air is



not sufficient to burn up all the gases formed. These are all compounds of hydrogen and carbon and in this zone they are partially consumed, burning to CO_2 and water, but a large part of the carbon is merely heated white hot thus rendering the flame luminous. Outside of the luminous zone is an almost invisible coat where the gases are completely consumed. The ultimate products of all the gases burned are carbon dioxid and water.

II. Press down upon a candle flame a piece of white letter paper or some other heavy paper until it almost touches the wick. Observe the charred ring with a white center. Cause? Which is the hottest part of the flame?

III. Hold a thin wire, either iron or platinum, across a candle flame or luminous burner flame. Note where the wire is hottest. Repeat with a piece of wire gauze. Repeat with a match stub or piece of wood.

B. NATURE OF FLAME.

I. Blow out the candle, then ignite the smoke. Why does it ignite?

II. Place a piece of wire gauze on a ring stand and turn on the gas in a burner placed under it. Now above the gauze light the gas. It will burn for some time above the gauze. Turn off the gas and allow the gauze to cool. Again light the gas, this time under the gauze. Why does the flame not pass through the gauze?

III. Heat a small glass tube until quite warm and at once place the end in the dark center of the candle flame and ignite the gas at the other end of the tube. What does this show?

C. LUMINOSITY OF FLAME.

I. Stop the holes of the Bunsen burner and note the effect. Why is this? Compare color of flames when the

holes are open and when they are shut. Take a burner apart and notice its construction. What is the use of allowing the air to enter the lower part of the tube and thus mix with the illuminating gas in it?

Rub two pieces of charcoal together over the blue flame and note the effect.

II. Press a cold spoon down on both burner flames and note effect. What makes the flame luminous? Which is the hotter of the two flames? Why?

D. CHEMISTRY OF FLAME.

I. Hold a candle in a wide mouthed bottle till it goes out.

II. Test the contents of the bottle by shaking up with lime water. One cc. is enough.

III. Hold a cold clean bottle over a luminous flame and note the deposit.

E. BLOWPIPE FLAME.

Close the holes in the Bunsen burner by turning the tube. Obtain the oxidizing flame by placing the blowpipe tip inside the dark portion of the flame just over the top of the burner, and blow across the flame somewhat strongly. An almost colorless pointed flame is produced. The reducing flame is obtained by placing the tip a little higher above the burner top and outside the flame. A luminous flame is produced. Which is the hotter? Why? When making these two flames do not blow so hard as to make much noise.

In the above Richter's 8th edition has been substantially followed.

METALS.

PRINCIPLES.

In the following statements the term salt includes only cases where the metal acts as a base, e. g., chromium salts include $CrCl_3$ not K_2CrO_4.

1. **Hydroxids** when brought in contact with acids form salts, provided they can be formed by any means in the presence of water. The same is true of oxids, except that ignited chromic oxid does not dissolve in acids.

2. All **nitrates, chlorates,** and **acetates** are soluble, but all salts of bismuth, tin, antimony, and the oxysalts of mercury, require some free acids to hold them in solution.

3. All **oxids** and **hydroxids** are insoluble, except those of the alkalis, those of the fourth group slightly soluble. The fixed alkalis precipitate solutions of all other metallic salts, the fourth group incompletely. The precipitate with silver, antimony and mercury is an oxid, in all other cases a hydroxid. This precipitate redissolves in eight cases, forming, if potassium hydroxid be used . . . K_2PbO_2, K_2SnO_2, K_2SnO_3, $KSbO_2$, $KSbO_3$, K_2ZnO_2, $KAlO_2$, $KCrO_2$. The latter reprecipitates on boiling.

4. **Ammonium hydroxid** precipitates solutions of the first three groups, manganese and magnesium imperfectly and not at all if ammonium chlorid be present. The precipitate is a hydroxid, except that with silver and antimony it is an oxid, with lead a basic salt, and with mercury

a peculiar salt. The precipitate redissolves in six cases, viz., silver, copper, cadmium, cobalt, nickel, and zinc. With silver NH_4AgO is formed, with zinc $(NH_4)_2 ZnO_2$.

5. The **chlorids** of the first group are insoluble, lead chlorid slightly soluble. **Hydrochloric acid** and **soluble chlorids** precipitate solutions of the salts of the first group, lead salts incompletely. (For higher oxids, see Prescott & Johnson, p. 251).

6. The **bromids** of lead, silver, and mercury are insoluble, mercuric bromid slightly soluble. **Hydrobromic acid** and **soluble bromids** precipitate solutions of lead, silver, and mercury, mercuric salts incompletely. (For higher oxids, see page 262, P. and J.)

The **iodids** of lead, silver, mercury, and cuprosum are insoluble. **Hydriodic acid** and **soluble iodids** precipitate solutions of lead, silver, mercury, and cuprosum. Cupric salts are precipitated as cuprous iodid with liberation of iodin. Ferric salts are merely reduced to ferrous salts with liberation of iodin.

8. The **sulfates** of lead, mercurosum, barium, strontium, and calcium are insoluble, those of calcium and mercurosum slightly soluble.

Sulfuric acid and **soluble sulfates** precipitate solutions of lead, mercurosum, barium, strontium, and calcium; calcium and mercurosum incompletely.

9a. The **sulfids** of the first three groups are insoluble. **Hydrosulphuric acid** transposes salts of the first two groups in acid, neutral, and alkaline mixtures.

Ferric solutions are reduced to ferrous with liberation of sulfur.

9b. **Soluble sulfids** transpose salts of the first three groups. The result is a sulfid, except that with aluminum and chromium salts it is a hydroxid, hydrogen sulfid being

evolved. With mercurous salts, mercuric sulfid and mercury are formed; with ferric salts, ferrous sulfid, and sulfur.

10. The **carbonates** of the **alkalis** are soluble.

(Condensed from Prescott & Johnson.)

DEFINITIONS.

A *precipitate* is a substance insoluble in the liquid which is present with it.

Filtration is the process of separating the precipitate from the liquid in which it is held, by pouring the whole on a filter paper in a funnel. The liquid runs through but the precipitate remains on the paper. The liquid is called the *filtrate.*

Decantation is the process of pouring off the liquid which holds a precipitate after allowing the precipitate to settle to the bottom of the vessel.

Reagent is the name applied to any known substance which is used in making an analysis.

Analysis is divided into two classes, Qualitative and Quantitative. A *qualitative* analysis is the name applied to the process of finding out what an unknown substance contains. *Quantitative* analysis proceeds further and determines the quantity of each ingredient.

GROUPING OF THE METALS.

The metals are grouped for purposes of analysis into five groups according as they are precipitated by certain reagents. The reagent which precipitates a whole group is called a group reagent.

I. Metals precipitated by hydrochloric acid; Lead, Mercury (monad), Silver.

Of these metals the chlorids are insoluble in water except lead chlorid which is slightly soluble.

II. Metals precipitated by hydrogen sulfid.

These precipitates are insoluble in cold dilute acids but readily soluble in warm.

Mercury (diad), Lead, Bismuth, Cadimum, Arsenic, Tin and Antimony belong to this group.

III. Metals not precipitated by the two first group reagents, but precipitated by ammonium hydroxide (NH_4OH) and hydrogen sulfid in the presence of ammonium chlorid. The ammonium chlorid is used to prevent the precipitation of magnesium, a fifth group metal. Iron, Chromium, Aluminum, Cobalt, Nickel, Manganese and Zinc belong to this group.

IV. Metals not precipitated by the reagents of groups one, two and three, but by ammonium carbonate $(NH_4)_2CO_3$ in presence of NH_4OH and NH_4Cl. Barium, Calcium and Strontium belong here.

V. Group V contains all metals not in the other groups. They are not precipitated by any of the above mentioned reagents. Fifth group metals are Magnesium, Potassium, Sodium and Ammonium (NH_4.)

LEAD.

SYMBOL Pb. ATOMIC WEIGHT 205.4.

GALENA.

A. 1. Examine the specimen as to,

a. Form.

b. Hardness.

c. Malleability.

d. Marking on paper.

2. Heat specimen on charcoal alone with the blow pipe.

Note: a. Color of coat (the deposit on the charcoal) hot and cold.

b. Distance of coat from assay piece.

c. Effect of *Reducing* flame on the coat. (Azure blue.)

d. Effect of oxidizing flame on coat.

Use a very small piece of galena, a piece not larger than a pin head.

3. Obtain the bead. (The little globule of metal.) Is it malleable? Easily cut? Will it mark paper? Will the finger nail scratch it?

4. Heat a small piece of galena on charcoal with sodium carbonate Na_2CO_3, note the coats as above. Do you get a malleable bead as before? What is this bead? Where has the sulfur gone to? Write the equation.

5. To make a solution. Try

I. Water cold and hot.

II. Hydrochloric acid cold and hot.

III. Nitric acid cold and hot.

Reaction. $3\ Pb + 8\ HNO_3 = 3\ Pb(NO_3)_2$ (lead nitrate) $+ 2\ NO + 4\ H_2O$.

NOTE.—After dissolving a metal in acid it is necessary to evaporate the solution nearly to dryness before testing. This removes any excess of acid. The solution may be diluted with water after evaporation to make a quantity great enough to work with.

B. Repeat the foregoing experiments with commercial lead.

C. Mix the solution from galena with that made from lead and test as follows.

To a small quantity of the solution in a test tube add a few drops of hydrochloric acid, note the precipitate and write the reaction. Repeat with H_2S, with H_2SO_4,

$K_2Cr_2O_7$, (potassium dichromate) and KI (potassium iodid). Tabulate your results and write out the reactions as follows.

Solution. $Pb(NO_3)_2 + HCl = \underset{\text{WHITE}}{PbCl_2}$ (lead chlorid) + ?

" $+ H_2S = PbS +$?

" $+ H_2SO_4 = PbSO_4$ (lead sulfate) + ?

" $+ K_2Cr_2O_7 = PbCrO_4 + CrO_3 + 2\ KNO_3$.

" $+ KI = PbI_2 +$?

SILVER.

SYMBOL AG. ATOMIC WEIGHT 107.11.

I. Heat a specimen of silver ore on charcoal alone in the reducing flame.

a. Obtain a bright bead.

b. Note color of the coat.

c. Heat a very long time in the reducing flame. Note a slight dark red coat.

II. Heat on charcoal with sodium carbonate in the reducing flame.

a. Obtain the bead.

b. Note time required for reduction as compared with I. What purpose does the carbonate serve?

III. Solution. Try

a. Water cold and hot.

b. Dilute nitric acid cold and hot.

c. Concentrated nitric acid cold and hot.

Evaporate the solution to expel an excess of acid and test after diluting with water as follows.

Solution $+ HCl = AgCl$ (silver chlorid) + ?

The white precipitate of the above + warm NH_4OH goes into solution once more with formation of $(NH_3)_3 (AgCl)_2$ (ammonio silver chlorid).

Solution + $FeSO_4$ = a black or dark precipitate of silver.

Sol. + $H_2S = Ag_2S$ + ?

Sol. + KI = AgI + ?

Finish up as many of the equations as you can and write the equation for solution of silver in nitric acid. If you have difficulty in doing this last perform the experiment once more and notice what gas is given off.

MERCURY.

SYMBOL HG. ATOMIC WEIGHT 198.49.

The symbol comes from the Latin name of mercury, hydrargyum. It occurs chiefly in the form of its sulfid (HgS) called cinnabar. Mercury occurs in two forms, as a monad, that is combining with or replacing one atom of hydrogen; also as a diad, combining with or replacing two atoms. It forms two different classes of salts. The chlorid of the monad is insoluble in water while the diad ($HgCl_2$) is easily soluble. The first is non-poisonous and is much used in medicine under the name of calomel. The other is the very poisonous corrosive sublimate.

A. Cinnabar (HgS).

1. Note the color of a large piece.
2. Powder, then note color again.
3. Heat on charcoal alone. It volatilizes.
4. Heat on charcoal with sodium carbonate in the reducing flame.
5. Heat in a glass tube with carbonate and note mirror like deposit on the cold walls of the tube.

B. Solution.

1. Put three parts of hydrochloric acid in one test tube and two parts nitric in another. Now put a little powdered cinnabar in each and note the effect.

2. Pour the contents of the two tubes together and note effect. Evaporate under the hood, dilute with water and test as under 3.

Reaction. When nitric and hydrochloric acids are brought together chlorin and an oxychlorid of nitrogen are set free. The chlorin when first made or nascent is a very active agent and dissolves mercury quickly, forming mercuric chlorid. Mercurous nitrate is formed sometimes when the solution is not heated.

$$3\ HCl + HNO_3 = \left\{ \begin{array}{c} NOCl_2 + Cl \\ \text{or} \\ NOCl + Cl_2 \end{array} \right\} + 2\ H_2O.$$

At the same time that the mercury is dissolved the sulfur of the cinnabar is changed into sulfuric acid. Test your solution for sulfuric acid with barium chlorid.

3. Mercury sol. $+ HCl = \underbrace{HgCl}_{\text{WHITE}}$ or nothing.

" " $+ NH_4OH = \underbrace{NH_2Hg_2Cl}_{\text{BLACK}} + NH_4Cl + 2\ H_2O.$

" " $+ KI =$ crimson or greenish HgI_2 or HgI.

" " $+ H_2S = HgS.$

After testing the solution you have made try a solution of some monad mercury compound and of some diad. Which have you made?

SEPARATIONS IN GROUP I.

Mix a solution of mercury with one of lead and silver. (This may be done by the instructor) Now to a quantity of the solution add HCl slowly, shake and allow to settle. Add more till no more precipitate is formed Filter.

$PbCl_2$.	$AgCl$.	$HgCl$.

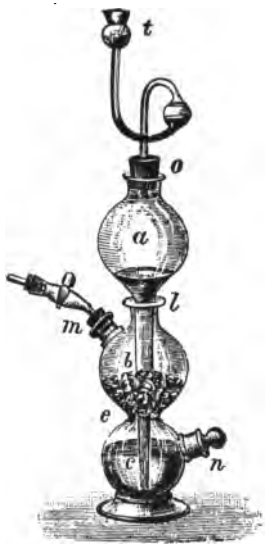
I. *Precipitate.*—Place on a filter and wash with a little distilled water. Now pour about twenty-five cc. boiling water on the precipitate and collect the solution in another test tube. Save the undissolved precipitate and test the filtrate by table 1. The hot water dissolves the lead chlorid so the hot solution contains lead if any is present.	Table 1. LEAD PB. Sol + H_2SO_4 = $PbSO_4$ (WHITE.) " + $K_2Cr_2O_7$ = $PbCrO_4$ (YELLOW.) " + KI = PbI_2 (YELLOW.) " + H_2S = PbS (BLACK.) See article on lead. Write out all the equations.
II. Pour warm ammonia on the undissolved precipitate and the silver chlorid will be dissolved, forming ammonio silver chlorid $(NH_3)_3(AgCl)_2$. See article on silver. Filter and test the filtrate by table 2.	Table 2. SILVER AG. Sol + H_2S = Ag_2S (BLACK.) " + KI = AgI (LIGHT YELLOW.) " + K_2CrO_4 (potassium chromate) = Ag_2CrO_4 (RED.)
III. If a black residue remains from II. mercury is indicated. Dissolve in nitrohydrochloric acid, dilute with water and test by table 3. If no residue is left of course this last is unnecessary.	Table 3. MERCURY HG. Sol + H_2S = HgS (BLACK.) " + KI = HgI (CRIMSON.)

Use as small quantities of reagents as possible in all these tests.

METALS OF THE SECOND GROUP.

CLASS I.

These may be divided into two classes. I. Includes all metals whose sulfids are soluble in yellow ammonium sulfid $(NH_4)_2S_x$. The sulfid may be made thus: Run H_2S from a Kipp's apparatus into ammonia until the ammonia smells strongly of the sulfid. To form the yellow variety allow this to stand or else heat a quantity of it with sulfur. Write out all the equations involved in this process. The metals of this subdivision are arsenic, antimony and tin.



ARSENIC.

SYMBOL As.

ATOMIC WEIGHT 74.44.

I. Make a solution of arsenic by dissolving some of the oxid As_2O_3 in hot water. This makes a good solution to test.

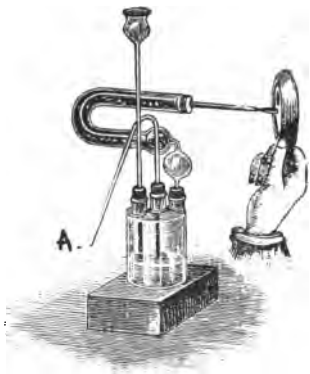
Sol + H_2S = $\underbrace{As_2S_3}_{\text{YELLOW.}}$ (arsenious sulfid) + ?

Sol + $AgNO_3$ (a single drop) = Ag_3AsO_3 + ?

Sol + $CuSO_4$ = $\underbrace{CuHAsO_3}_{\text{GREEN.}}$ (Scheele's green). When copper acetate is used instead of the sulfate the well known Paris green is formed. $(CuOAs_2O_3)_3Cu(C_2H_3O_2)_2$.

II. Marsh's test for arsenic. (Under hood). As this experiment is somewhat dangerous for a novice it is better to perform it as a teacher's experiment. Make a hydrogen generator as described under hydrogen from a wide-mouthed bottle or a three-necked Wolff's bottle as

shown in the figure. A, is used merely as a safety tube. Chemists use various modifications of this apparatus, as will be seen by consulting different text books, but that here figured and described will answer the purpose well.



Put granulated zinc and sulfuric acid or hydrochloric acid in the generator as described under hydrogen, and when the gas is free from air light a small jet of it. *Observe all possible precautions to prevent explosions.* Now hold a piece of cold white porcelain in the flame. If the zinc is pure there should be no spot left on the porcelain. Blow out the flame and pour into the generator a few drops of some arsenic solution. When absolutely free from air light again. Arsin (AsH_3) is formed according to the equation given below and on burning in the air forms metallic arsenic, which will be deposited on the cold porcelain.

Reaction. $H_3AsO_3 + 3\ Zn + 3\ H_2SO_4 = 3\ ZnSO_4$ (zinc sulfate) $+ 3\ H_2O + AsH_3$ (arsin).

This experiment should never be performed except under a hood, as the fumes of arsin are *extremely* poisonous.

III. Make several arsenic spots on porcelain. Touch one with a drop of hot nitric acid. It dissolves. Try HCl. It is insoluble. Try with a drop of bleaching powder solution. It is soluble. This same test is applied for antimony, but a spot of antimony is insoluble in a solution of bleaching powder.

ANTIMONY.

SYMBOL SB. ATOMIC WEIGHT 119.52.

The symbol Sb comes from the Latin name of antimony, stibium.

I. Make a solution of antimony by dissolving the metallic antimony in nitrohydrochloric acid. Try also hydrochloric acid. Do not evaporate this time.

II. To a small portion of the hydrochloric acid solution add some water. A white powder is precipitated. This is characteristic of antimony and bismuth. A white basic chlorid is formed.

Solution + H_2S = Sb_2S_3 (antimonius sulfid). This is of a bright orange color and this test is more often used for antimony than any other.

III. Try the Marsh's test for antimony. Obtain the spots. Compare with arsenic spots. Test with a bleaching powder solution. The spots do not dissolve. Try to dissolve a spot in hot nitric acid. How can you tell the difference between spots of arsenic and antimony?

The antimony solution with hydrochloric acid and zinc forms a gaseous compound called stibin (SbH_3), which is much like arsin though not so poisonous.

TIN.

SYMBOL SN. ATOMIC WEIGHT 118.15.

The symbol comes from the Latin name stannum.

I. Examine a piece of metallic tin. Bend the piece noting the crackling sound. This sound is very characteristic of tin. It is supposed that a piece of tin is composed of a great number of minute crystals or granules, which rub upon one another when the piece is bent.

II. Solution. Try HCl cold and hot. Test the gas generated with a flame. What is it? Write the equation knowing that stannous chlorid ($SnCl_2$) is formed.

Solution + $HgCl_2$ (mercuric chlorid) = $\underset{\text{BLACK}}{\underline{Hg}}$ + $SnCl_4$

$SnCl_4$ is stannic chlorid. This test is used to determine both mercury and tin. Note that before the black mercury is precipitated a white powder is formed. This is HgCl. Compare with the analysis of first group metals.

Solution + H_2S = $\underset{\text{BROWN}}{\underline{SnS}}$ (Stannous sulfid.) Treat a bit of this sulfid with HCl. What is formed? Write the equation.

Sol + NH_4OH + a zinc strip = a deposit of metallic tin on the zinc. The ammonia is used to make the solution alkaline.

The metals of this first class may be easily distinguished from each other by the color of the precipitates which they form with hydrogen sulfid.

METALS OF THE SECOND GROUP.

CLASS 2.

The sulfids of this class are insoluble in yellow ammonium sulfid. It contains lead, copper, cadmium, bismuth and the diad mercury. Lead occurs in this group as well as in the first because it is but imperfectly removed by HCl.

MERCURY (DIAD).

See discussion of mercury under group I.

NOTE. When a diad mercury salt is precipitated by H_2S, first a greenish precipitate is formed which gradually changes to black.

COPPER.

SYMBOL Cu. ATOMIC WEIGHT 63.12.

Symbol comes from the Latin name cuprum.

COPPER OXID CuO.

I. Examine specimen. Note color, form, weight, etc.

II. Heat on charcoal in the reducing flame alone.

III. Heat on charcoal in the reducing flame with sodium carbonate. Examine the bead thus formed. Color, etc. You have metallic copper.

IV. Heat a mixture of equal parts of powdered charcoal or lamp black and copper oxid in a small glass tube. What is formed? What becomes of the oxygen of the copper oxid? Answer experimentally. Compare copper thus formed with a metallic speciman.

V. Solution. Try HCl cold and hot, HNO_3 cold and hot. What gas is formed in the latter case? Write the equation. Note the color of the solution. Note the color of all the copper solutions in the laboratory that you can find. $CuSO_4$, $Cu(NO_3)_2$ $Cu(C_2H_3O_2)_2$, $CuCl_2$, etc. What seems to be the characteristic color.

Try concentrated sulfuric acid cold then hot. *Be very careful* in handling hot concentrated sulfuric acid as it is a very dangerous body. Note the odor of the gas thus formed. Where has this same reaction been used before? Write the equation.

VI. Use the nitric acid solution for the following tests after having first freed it from excess of acid.

You have a solution of $Cu(NO_3)_2$ (copper nitrate.)

Sol + an iron wire = metallic Cu.

Sol + NH_4OH = $(N_2H_6Cu)O(NH_4NO_3)_2$ a deep blue solution called cuprammonium nitrate.

$$Sol + H_2S = \underset{\text{BLACK.}}{\underline{CuS}}$$

$$Sol + K_4Fe(CN)_6 \text{ (potassium ferrocyanid).}$$
$$= \underset{\text{REDDISH BROWN.}}{\underline{Cu_2Fe(CN)_6}} \text{ (copper ferrocyanid).}$$

Finish all these equations.

VII. Dip a platinum wire in any copper solution, then hold it in the colorless flame of a Bunsen burner. Note color given to flame. Have we studied any other elements which might be confounded with Cu in this test?

VIII. Bend a small loop in a piece of platinum wire and heat it in the colorless flame of a burner to redness. Plunge it while hot into a bottle of powdered borax and heat again until a clear bead is obtained. Dip the bead in a copper solution and heat with the blowpipe, using the oxidizing flame. The bead will be green while hot and blue when cool. Make this test several times so as to become skilful.

CADMIUM.

SYMBOL Cd. ATOMIC WEIGHT 111.10.

Cadmium is quite rare. If a soluble salt of the metal can be obtained make a solution and precipitate the cadmium with hydrogen sulfid. Note color of precipitate. It is cadmium sulfid (CdS.) Write the equation.

SEPARATIONS IN GROUP II.

CLASS I.

Make a solution containing several first and some second group metals and try to separate them according to the following scheme.

I. After all the first group has been removed by hydrochloric acid, the second group remains. Now to the filtrate from group one add hydrogen sulfid until no more

precipitate is formed. The precipitate may contain As_2S_3 Sb_2S_3, SnS, PbS, HgS, CdS, CuS or Bi_2S_3.

II. Dissolve out sulfids of class 1, by heating the whole precipitate with yellow ammonium sulfid in a test tube.

III. Filter and save precipitate for class 2. Neutralize the ammonia of the ammonium sulfid with hydrochloric acid and the precipitates of arsenic, antimony and tin are thrown down again. You can see whether one metal is present or several by the color of the sulfid. If the color is a clear yellow, arsenic is indicated (page 97); if orange, antimony (page 101); if brown, tin (page 103). But if the color is indistinct there may be a mixture of these metals present and the Marsh's test must be resorted to.

CLASS 2.

Contains the Sulfids of Mercury, Lead, Bismuth, Copper, and Cadmium.

1. Wash the precipitate which remains undissolved in ammonium sulfid and water.
2. Dissolve the precipitate in hot dilute nitric acid. Expel excess acid and dilute with water.

NOTE.—The sulfids of lead, bismuth, copper and cadmium dissolve in HNO_3. HgS does not.

I. RESIDUE HgS.

Dissolve the black precipitate in a little nitro-hydrochloric acid. Expel the excess of acid and dilute with water. Test for mercury with:

1. Potassium iodid gives a red precipitate HgI_2.
2. Stannous chlorid = a black precipitate of metallic mercury. (See the article on tin).

II. SOLUTION: *May contain* $Pb(NO_3)_2$, $Bi(NO_3)_3$, $Cu(NO_3)_2$ *or* $Cd(NO_3)_2$

To a portion of the solution add a drop of dilute sulfuric acid. If there is a precipitate add to the whole solution, warm and filter. This removes the lead.

PRECIPITATE.
Lead.

To confirm for lead, test some of the precipitate by heating on platinum foil or in a crucible with potassium chromate or dichromate. Yellow lead chromate is formed $PbCrO_4$.

III. FILTRATE from II: *May contain* $Bi(NO_3)_3$, $Cu(NO_3)_2$, *or* $Cd(NO_3)_2$.

Add ammonia to neutralize acid and precipitate bismuth. Copper and cadmium remain in solution.

IV. PRECIPITATE: $Bi(OH)_3$. (WHITE)

Filter and wash precipitate and test by potassium stannite K_2SnO_2 thus. Add potassium hydroxid (KOH) to a solution of stannous chlorid until the precipitate first formed dissolves again. Potassium stannite is formed which when added to the $Bi(OH)_3$ will turn the precipitate black with formation of dibismuth dioxid Bi_2O_2.

V. SOLUTION: $Cu(NO_3)_2$ *or* $Cd(NO_3)_2$.

When the bismuth is precipitated if copper is present blue cuprammonium compounds will be formed. (See copper). To confirm, test solution on a platinum wire.

VI. CADMIUM.

Copper and cadmium may have been present together. From the ammonia solution precipitate both again with hydrogen sulfid. Dissolve the precipitate in hot dilute H_2SO_4. Cadmium sulfid dissolves but copper sulfid does not. Precipitate the solution once more with hydrogen sulfid. A yellow precipitate CdS will be formed if cadmium is present.

THIRD GROUP METALS.

The metals of the third group are also divided into two classes, A and B. Class A includes metals which are precipitated by NH_4OH in the presence of NH_4Cl. Such are chromium, iron and aluminum.

To class B belong those metals not precipitated by NH_4OH and NH_4Cl, but by ammonium sulfid $(NH_4)_2S$. Cobalt, nickel, manganese and zinc belong to this class.

IRON.

SYMBOL FE. ATOMIC WEIGHT 56.

The symbol comes from the Latin name *ferrum*. Iron forms two classes of salts, ferrous or diad and ferric or triad salts. The diad salt is very easily converted into the triad by exposure to the air, so that nearly all diad compounds will give tests for the triad or ferric compounds.

Solution. Iron has several solvents, but its action is somewhat peculiar with each. Note what is formed in each of the following cases.

I. Dissolve some iron wire in HCl. What gas is set free? $FeCl_2$ is formed which rapidly changes to $FeCl_3$.

II. Try H_2SO_4 cold, then hot. In which case is the action most rapid? What gas is formed? Where have we seen this reaction before? Ferric sulfate is formed. Write the complete equation.

III. Try hot dilute nitric acid. Note gas formed. What other metals set free this same gas on solution in HNO_3? The ferric salt is produced. Write the equation. You now have solutions of ferrous and ferric salts. Test them according to the following:

DISTINCTION BETWEEN FERROUS AND FERRIC IRON.

1. Ferric compounds.
   a. Potassium ferricyanid gives no precipitate, but a green color.
   b. Potassium sulfocyanid gives a blood red precipitate.
   c. Potassium ferrocyanid gives a blue precipitate.
2. Ferrous compounds.
   a. Ferricyanids give a deep blue precipitate.
   b. Sulfocyanids give no change.
   c. Ferrocyanids give a pale blue precipitate.

Note the difference in the color between ferrous and ferric compounds.

Equations:

1. Ferric compounds.
   a. $FeCl_3 + K_3Fe(CN)_6 = o.$
   b. $2\ FeCl_3 + 6\ KCNS = Fe_2(CNS)_6 + ?$
   c. $4\ FeCl_3 + 3\ K_4Fe(CN)_6 = Fe_4(Fe(CN)_6)_3 + 12\ KCl.$
2. Ferrous compounds.
   a. $3\ FeSO_4 + 2\ K_3Fe(CN)_6 = Fe_3Fe_2(CN)_{12} + 3\ K_2SO_4.$
   b. $FeSO_4 + KCNS = o.$
   c. $FeSO_4 + K_4Fe(CN)_6 = K_2FeFe(CN)_6 + K_2SO_4.$

CHROMIUM.

SYMBOL CR. ATOMIC WEIGHT, 52.

The metal chromium is quite rare. It is most familiar to us in the form of some of its salts, as chrome alum ($K_2Cr_2(SO_4)_4 + 24\ H_2O$), Potassium chromate ($K_2CrO_4$), potassium dichromate ($K_2Cr_2O_7$), etc.

I. Make a solution of $K_2Cr_2O_7$ in water by dissolving one part of the salt in about ten or fifteen parts of water.

II. Add to a small portion a few drops of $AgNO_3$ solution. A red precipitate of Ag_2CrO_4 is formed.

III. Add H_2S to a little of the $K_2Cr_2O_7$ solution. The color changes from yellow or orange to green. Very characteristic of chromium. Add lead acetate $Pb(C_2H_3O_2)_2$, you obtain as a yellow precipitate lead chromate $PbCrO_4$.

ALUMINUM.

SYMBOL AL. ATOMIC WEIGHT, 27.

Aluminum is best known to us in the compound, $KAl(SO_4)_2 + 12\ H_2O$, common alum, and, in the metallic form.

It is a white metal of very low specific gravity.

I. Make a solution by dissolving the metal in HCl or a piece of alum in water.

II. Tests;

$$\text{Sol} + Na_2HPO_4 = \underset{w}{\underline{Al_2(PO_4)_2}} + ?$$

$$\text{Sol or } Al_2Cl_6 + 6\ KOH = \underset{w}{\underline{Al_2(OH)_6}} + 6\ KCl.$$

$$Al_2(OH)_6 + 2\ KOH = K_2(AlO_2)_2 + 4\ H_2O.$$

Complete the equations according to solution used.

When a salt of aluminum is precipitated with a hydroxid $Al_2(OH)_6$, a white precipitate, is first formed. On addition of more hydroxid this dissolves to form $K_2(AlO_2)_2$ provided KOH was used. An addition of ammonium chlorid prevents the precipitate from redissolving. Compare potassium aluminate ($K_2(AlO_2)_2$) with potassium stannite (K_2SnO_2).

NICKEL AND COBALT.

These two metals are alike in many respects, and as they are nearly always found associated they may be considered together. Considerable difficulty is always experienced in separating them, hence it will be better for the beginner not to attempt it.

NICKEL.

SYMBOL Ni. ATOMIC WEICHT, 58.24.

I. Test a nickel salt on a borax bead with the oxidizing flame, you should obtain a bead which is brownish red while hot and yellow when cold.

II. Try the same with a cobalt salt. The bead will be blue.

III. Test a nickel solution with NH_4OH, at first a green precipitate of $Ni(OH)_2$ is formed. An excess of NH_4OH gives a blue solution.

$$\text{Sol} + (NH_4)_2S = \underbrace{NiS}_{\text{BLACK.}}$$

$$\text{Sol} + KOH = \underbrace{Ni(OH)_2}_{\text{GREEN.}}$$

Complete the equations.

IV. Dissolve a five cent piece in nitric acid and find out what it contains.

COBALT.

SYMBOL Co. ATOMIC WEIGHT, 58.49.

I. $\text{Cobalt sol} + (NH_4)_2S = \underbrace{CoS}_{\text{BLACK.}}$

$\text{Cobalt sol} + KOH = \underbrace{Co(OH)_2}_{\text{BLUE.}}$

$\text{Cobalt sol} + Na_2CO_3 =$ a red basic carbonate of cobalt, which when boiled produces a violet or blue solution.

MANGANESE.

SYMBOL MN. ATOMIC WEIGHT, 54.57.

The chief sources of manganese are the ores Rhodochrosite ($MnCO_3$), Manganite ($Mn_2O_2(OH)_2$), Pyrolusite (MnO_2), etc.

I. Dissolve a very small quantity of manganese dioxid (MnO_2) in hydrochloric acid. What gas is set free? Write the equation for the reaction. You now have a solution of manganese chlorid ($MnCl_2$). Expel excess of acid and dilute.

II. Into a quantity of $MnCl_2$ in a test tube run hydrogen sulfid. What happens? To the solution containing H_2S add ammonia. Note change. To a manganese solution add sodium carbonate.

Reactions:

$$MnCl_2 + H_2S = o.$$

$$MnCl_2 + (NH_4)_2S = \underbrace{MnS}_{\text{PINK.}} + ?$$

$$MnCl_2 + Na_2CO_3 = \underbrace{MnCO_3}_{\text{WHITE.}} + ?$$

III. Boil a small quantity of manganese chlorid solution with nitric acid and lead dioxid (PbO_2) or red lead (Pb_3O_4). When the sediment subsides a purple solution will be seen. This is permanganic acid ($H_2Mn_2O_8$).

IV. Heat a small quantity of MnO_2 in a borax bead in the oxidizing flame of the blow pipe. A violet bead will be obtained which will slowly change to red when cool. Heat the red bead in the reducing flame and the color will disappear. This red color is caused by manganic oxid, which is the coloring matter of several minerals, such as amethyst, etc.

ZINC.

SYMBOL ZN. ATOMIC WEIGHT 64.91.

Zinc never occurs free in nature. It is found usually as a silicate in the mineral Willemite ($ZnSiO_4$), as a sulfid in zinc blende (ZnS), as a carbonate in Smithsonite ($ZnCO_3$), etc.

I. Dissolve a piece of zinc in hydrochloric acid. What gas is formed? What salt of zinc have you? Try the same with sulfuric acid. What is formed this time?

II. To a solution of some zinc salt add H_2S. No precipitate should be formed. To the solution containing H_2S add ammonia. A white precipitate of zinc sulfid (ZnS) is formed. Write the reaction. (Hydrogen sulfid has no action on metals of the third group, but when an alkali such as ammonia is present an alkaline sulfid is formed which precipitates the metals).

NOTE.—Either of the solutions made in I. when freed from excess acid may be used in this experiment.

III. To a zinc solution add a solution of sodium carbonate, zinc carbonate ($\underbrace{ZnCO_3}_{\text{WHITE}}$) is formed. Write out all the equations for the above.

IV. On a piece of charcoal heat a very small piece of zinc with sodium carbonate in the oxidizing flame. A yellow coating of zinc oxid is formed which changes to white on cooling. Moisten this coating with a solution of cobaltous nitrate. The coat is changed to green.

SEPARATIONS IN GROUP III.

May Contain Iron, Chromium, Aluminum, Nickel, Cobalt, Manganese and Zinc.

To the clear filtrate left from Group II, which has been freed from hydrogen sulfid by boiling, add a few

drops of nitric acid and boil to change ferrous iron to ferric if iron should be present. Add ammonium chlorid and ammonia until the solution smells strongly. If a precipitate is formed filter and wash.

PRECIPITATE: *Class A of Third Group. May contain ferric hydroxid* ($Fe(OH)_3$), *chromium hydroxid* ($Cr(OH)_3$), *aluminum hydroxid* ($Al(OH)_3$).

Pierce the point of the filter and wash the precipitate into a test tube, using as little water as possible. Add potassium or sodium hydroxid and boil a few minutes. If a precipitate remains, filter and wash.

PRECIPITATE: $Fe(OH)_3$ *or* $Cr(OH)_3$.		SOLUTION: $K_2Al_2O_4$.
For iron dissolve the precipitate in a small quantity of HCl and test the solution $FeCl_3$ for iron with KCNS. If iron is present the original solution must be tested to determine whether it is in the ferrous or ferric condition. (See iron). Remember that some ferric iron will always be present in ferrous solutions which have been exposed to the air.	*For chromium* test a portion of the precipitate by heating in a crucible or on platinum foil with potassium nitrate and sodium carbonate to change to chromate. Dissolve the potassium or sodium chromate thus formed in water. Add acetic acid($HC_2H_3O_2$)and test for chromium with lead acetate. (See article on chromium). NOTE.—When chromium is present it is indicated by a change in color of the original solution from yellow to green when treated with H_2S.	Potassium aluminate. See aluminum. *For aluminum* add to the potassium or sodium solution a very small quantity of HCl, just enough to make it acid, then add ammonium carbonate. A precipitate ($\underset{\text{WHITE}}{Al(OH)_3}$) indicates aluminum. See article on aluminum.

FILTRATE: *Class B. May contain zinc, nickel, cobalt or manganese.*

Add ammonium sulfid to the filtrate from Class A, and if a precipitate appears, warm until it settles. Filter and wash. Examine filtrate for the Fourth Group.

PRECIPITATE; *ZnS, NiS, CoS, or MnS.*

Treat on the filter with cold dilute HCl. Note if a black residue remains. Boil the filtrate to free from ammonium sulfid.

RESIDUE: *NiS or CoS, Black.*	SOLUTION: *$ZnCl_2$ or $MnCl_2$.*	
Test by the blowpipe for nickel or cobalt. (See nickel and cobalt, page 119).	Add an excess of potassium or sodium hydroxid and allow to stand for several minutes without heating. Filter.	
	PRECIPITATE: *$Mn(OH)_2$.*	SOLUTION: *K_2ZnO_2.*
	Oxidize to permanganic acid as described under manganese(page 121). Test by blowpipe.	Precipitate again with ammonium sulfid. A white precipitate ZnS indicates Zn. Study the reactions given under Zn.

METALS OF GROUP IV.

The metals of this group are Barium, Strontium, and Calcium. They rapidly oxidize on exposure to the air, and so never are found free in nature.

BARIUM.

SYMBOL BA. ATOMIC WEIGHT 136.

Barium is a silver white metal which is very ductile and malleable. As a metal it is extremely hard to obtain. It occurs in the form of ore, as Heavy Spar, ($BaSO_4$), and as Witherite, ($BaCO_3$).

I. Make a solution of $BaCl_2$ (barium chlorid) in water. Dip into the solution a platinum wire and hold in the colorless flame of the burner. Note color given to flame. What other elements have we studied that have given a like color to the flame?

II. To a small quantity of a barium solution add a few drops of dilute H_2SO_4. Note precipitate. Write the equation. Where has this reaction been used before? Is $BaSO_4$ soluble in water?

III. Barium solution $+ K_2CO_3 = BaCO_3 + ?$ White precipitate.

IV. Barium solution + K_2CrO_4 = $BaCrO_4$ + ? or Barium solution + $K_2Cr_2O_7$ = $BaCrO_4$ + ? Yellow precipitate.

STRONTIUM.

SYMBOL SR. ATOMIC WEIGHT 87.

Strontium occurs in strontianite ($SrCO_3$), and in celestite ($SrSO_4$). It is much like barium, being a white or light yellow metal, malleable and ductile. It never occurs as a metal, however, and is rapidly oxidized.

I. Test a strontium solution with platinum wire. It colors the flame crimson. This is the easiest and most general test for strontium.

II. Test a strontium solution with H_2SO_4. $SrSO_4$ is precipitated. Try to dissolve in hot water. Is it more or less soluble in water than $BaSO_4$?

CALCIUM.

SYMBOL CA. ATOMIC WEIGHT 40.

Like barium and strontium, calcium never occurs free. It occurs chiefly in the form of its carbonate in marble, chalk, limestone, etc.; also as a fluorid in fluorspar (CaF_2), as a sulfate in gypsum ($CaSO_4$), as a phosphate in apatite ($Ca_3(PO_4)_2$), etc.

I. Dissolve a piece of marble ($CaCO_3$) in HCl. What gas is generated? What salt is formed? Evaporate nearly to dryness and thus make a very concentrated solution. Add a few drops of H_2SO_4. Note the precipitate. Is it soluble in water? How does $CaSO_4$ compare with $BaSO_4$ and $SrSO_4$ as to solubility in water?

II. Calcium solution + KOH = $Ca(OH)_2$ + ? White precipitate.

III. Precipitate a dilute solution of some calcium salt which contains free ammonia with ammonium oxalate

and allow the white precipitate to stand for several hours. Examine the precipitate with a microscope.

IV. Test a calcium solution with a platinum wire. It colors the flame brick red.

SEPARATIONS OF GROUP IV.

May contain barium, calcium or strontium.

To the filtrate from group III containing ammonium chlorid add ammonium carbonate and hydroxid. Heat gently for some time, then filter.

$BaCO_3$, $SrCO_3$ and $CaCO_3$ will be precipitated.

Dissolve the well washed precipitate in dilute acetic acid. The solution will contain acetates of barium, calcium and strontium. To a small portion of this solution add potassium dichromate and if a precipitate appears add the reagent till no more precipitate is formed then filter and wash.

PRECIPITATE: $\underset{\text{YELLOW.}}{\underline{BaCrO_4}}$

Confirm by dissolving this precipitate in HCl and precipitating again with H_2SO_4. White $BaSO_4$ is formed. Try also the flame test for barium.

SOLUTION: $Sr(C_2H_3O_2)_2$ *or* $Ca(C_2H_3O_2)_2$.

Precipitate with ammonium carbonate $(NH_4)_2CO_3$ with ammonium hydroxid. Filter and wash the precipitate then dissolve once more in dilute acetic acid and divide the solution into two portions.

I. FOR STRONTIUM.

To a portion add a solution of calcium sulfate; boil, allow to stand for a short time and a white precipitate $SrSO_4$ indicates strontium.

Test by platinum wire in the colorless flame. (See page 107). Crimson color is given the flame.

II. FOR CALCIUM.

Add a solution of potassium sulfate and filter to remove all strontium. To the filtrate add NH_4OH and ammonium oxalate $(NH_4)_2C_2O_4$. A precipitate of $\underset{\text{WHITE}}{\underline{CaC_2O_4}}$ indicates calcium. This precipitate is insoluble in $HC_2H_3O_2$ but soluble in HCl.

METALS OF GROUP V.

The metals of this group consist of Magnesium, Potassium, Sodium and Ammonium (NH_4). The last, ammonium, is not strictly a metal, but since it acts as such, replacing hydrogen in acids to form salts, etc., it is treated as a metal.

SEPARATIONS OF GROUP V.

I. Evaporate a drop or two of the original solution on a piece of platinum foil at a gentle heat. If a residue is obtained heat strongly and if this residue vaporizes quickly ammonium compounds are indicated.

FOR AMMONIUM.

Take a portion of the original material with potassium or sodium hydroxid and heat gently. The odor of ammonia may be noticed, or, it may be detected by its effect on moistened litmus paper, or, by the effect on a rod dipped in HCl.

Or, conduct the gas into water and add *Nessler's reagent, a brown precipitate of nitrogen dimercuric iodid, HHg_2I is produced.

Test another portion with $HgCl_2$. A white precipitate of nitrogen dihydrogen mercuric chlorid is produced. (NH_2HgCl.)

FOR MAGNESIUM.

Test the filtrate from group four.

Add a little ammonium hydroxid then enough ammonium chlorid to dissolve any precipitate which may appear. Now add sodium phosphate and a white precipitate $MgNH_4PO_4$ indicates magnesium.

If magnesium is present it will prevent the tests for sodium and potassium. So it must be removed. To remove magnesium, precipitate it with barium hydroxid, then remove the barium with sulfuric acid.

FOR POTASSIUM.

Test by the flame on platinum wire.

Violet color is given to the flame which is not hidden by blue glass.

FOR SODIUM.

Test in the flame. Yellow color is imparted to flame.

The color is obscured by blue glass

NOTE.—A sufficient number of pieces of cobalt glass should be placed at convenient points in the laboratory.

NOTE.—*Nessler's reagent is made by adding to a solution of mercuric chlorid a solution of potassium iodid until the precipitate is nearly all redissolved; then add solution of potassium hydroxid. Leave until the liquid becomes clear and use after decantation to free from any remaining sediment.

SUMMARY.

Grouping of the Metals.

I. Add HCl a drop at a time as long as any precipitate is produced. Warm, shake and filter.

Precipitate.

Group I.

Lead, $PbCl_2$. (WHITE.)

Silver, $AgCl$. (WHITE.)

Mercury (monad) Hg_2Cl_2.

Proceed as on page 95.

II. To the filtrate, or, if group I. is absent to the origin solution + HCl add hydrogen sulfid until the solution smells strongly of the gas. Warm and filter.

Precipitate.

Group II.

Arsenic, As_2S_3. (YELLOW.)

Antimony, Sb_2S_3. (ORANGE.)

Tin, SnS. (BROWN.)

Lead, PbS. (BLACK.)

Bismuth, Bi_2S_3. (BLACK.)

Copper, CuS. (BLACK.)

Cadmium, CdS. (YELLOW.)

Mercury, HgS. (BLACK.)

Changes from white and yellow to black.

III. Boil to expel excess of H_2S. Proceed by pages 125, 127.

Precipitate.

Group III.

A. By ammonium hydroxid and ammonium chlorid.

Aluminum, $Al_2(OH)_6$. (WHITE. Jelly like.)

Chromium, $Cr_2(OH)_6$. (BLUE GREEN.)

Iron, $Fe_2(OH)_6$. (BROWN.)

B. By ammonium sulfid.

Manganese, MnS. (PINK.)

Cobalt, CoS. (BLACK.)

Nickel, NiS. (BLACK.)

Zinc, ZnS. (WHITE.)

IV. To the filtrate from group III. containing NH_4OH and NH_4Cl add $(NH_4)_2CO_3$ and NH_4OH. Heat gently for some time. Do not boil. Filter.

Precipitate.

Group IV.

Barium, $BaCO_3$. (WHITE.)

Strontium, $SrCO_3$. (WHITE.)

Calcium, $CaCO_3$. (WHITE.)

TESTING FOR ACIDS.

Before testing, all the metals which would interfere may be removed by treating the unknown with a concentrated solution of potassium or sodium carbonate.

To a small quantity of the solid substance of the unknown or to a concentrated solution of the same add a little concentrated H_2SO_4 and heat gently. If the vapor reddens moist litmus paper some of the following acids may be present.

I. If there is effervescence, CO_2 is indicated and a carbonate is probably present.

II. If the gas has an odor it may be:

a. H_2S has odor of rotten eggs, and blackens paper moistened with lead acetate.

b. SO_2, sulfurous anhydrid. Odor of burning sulfur.

c. $H(C_2H_3O_2)$, odor of vinegar. Gives a red solution with ferric chlorid.

d. HCl, slight sharp odor. Forms a white cloud when brought in a contact with ammonia vapor.

III. Gases having color:

a. HI. Hydriodic acid. Violet color. Colors CS_2 blue or violet. See page 15.

b. HBr. Hydrobromic acid. Brown color. Test with CS_2. See page 15.

IV. $HClO_3$. Chloric acid. Explodes when substance is brought in contact with H_2SO_4. Has odor of chlorin.

V. For nitric acid try the brown ring test. See page 53.

VI. For sulfuric acid and sulfates, precipitate the original solutions with $BaCl_2$. $BaSO_4$ of a white color is formed.

Chlorin is sometimes evolved when hypochlorites, bleaching powder, etc., are present in the unknown.

SOLUTION.

I. Add to a test tubeful of hot water as much salt as it will dissolve. Allow to cool slowly and note the crystals formed.

Repeat with copper sulfate, potassium chlorate. Examine the crystals with a microscope or good magnifier. Detemine the system of crystallization if possible, referring to any work on mineralogy.

II. Determine the temperature of twenty cc. distilled water. Add a quantity of common salt, stir and again note the temperature. Try the same with sodium nitrate ($NaNO_3$) copper sufate, etc.

III. To a small quantity of water in a test tube gradually add concentrated sulfuric acid and note changes in temperature. Do you see why we should be careful in diluting concentrated sulfuric acid?

IV. In a small quantity of water in an evaporating dish place a small piece of potassium or sodium hydroxid, determining the the temperature change.

SOLUTION OF METALS, THEIR OXIDS AND HYDROXIDS IN ACIDS.

I. Dissolve a small piece of zinc in dilute hydrochloric acid. What gas is generated? Write the equations. Evaporate nearly to dryness, then take a drop of the solution, place on a microscope slide and allow to evaporate to dryness without further heating. This brings about the formation of good crystals.

Repeat same with iron filings, with magnesium wire.

Repeat with copper. What gas is formed? Why not hydrogen?

II. Repeat the above with zinc oxid, ferric oxid, magnesium oxid, and calcium oxid. What gas has been formed?

III. Repeat with the hydroxids of the above metals. Evaporate the various solutions to dryness, examine with the microscope and determine whether the salts formed are the same as when metals or their oxids are dissolved. Write out all the equations connected with these three experiments.

IV. Repeat the three above experiments with sulfuric acid instead of hydrochloric. Write all the equations.

NOTE.—Dilute the acid for this experiment, using one part of concentrated acid to nine of water.

WATER OF CRYSTALLIZATION.

I. Dry some crystals of common salt on filter paper and heat in a dry test tube. Is any water given off? Try the same with crystals of copper sulfate, with sodium sulfate, with zinc sulfate. How do these latter substances differ from common salt?

To the dry copper sulfate after cooling add a little water. Note change in color. Note change in temperature.

II. EFFLORESCENCE.—On three separate watch crystals place small crystals of, (a) sodium carbonate, (b) sodium sulfate, and (c) copper sulfate. Let them stand over night and note changes.

III. Repeat II, using calcium chlorid and potassium and sodium hydroxids.

DETERMINATION OF WATER OF CRYSTALLIZATION.

I. $CuSO_4$. Clean out very carefully a small porcelain crucible, dry, cool and weigh very exactly on a good pair of balances. Handle the crucible with a pair of pincers, as touching it with the fingers will make a variation in the results. Select a good clean crystal of copper sulfate weighing about two grams and place in the crucible. Weigh again the crucible + the copper sulfate. Of course the difference will be the exact weight of the sulfate. Place in an air bath and heat to 225° for an hour. At the end of the time place in a desiccator and allow to cool, then weigh again. The loss in weight represents the weight of water in copper sulfate. Calculate the per cent. of water thus, copper sulfate crystallizes with five molecules of water:

$CuSO_4 + 5\ H_2O : 5\ H_2O : : 100 : x$

where x = the wished per cent.

$64 + 32 + 04 + 90 : 90 : : 100 : x$

$x = 36$ per cent.

Calculate in the same way, placing the actual weight of the crystal in the place of $CuSO_4 + 5\ H_2O$ and the weight of water for $5\ H_2O$. Repeat the experiment several times till you can get *accurate* results.

NOTE.—Often a failure arises from heating too short a time, thus not freeing from water completely.

II. Repeat the above with barium chlorid $BaCl_2 + 2\ H_2O$, heating to a temperature of 125–150°.

III. a. Heat two or three grams of gypsum ($CaSO_4 + 2\ H_2O$) in a hot air bath to about 105°, until it will break up into a fine powder. You now have plaster of Paris. Make into a paste and watch it, "set."

b. Heat about three grams of gypsum, which has been carefully weighed, in an air bath to about 250° (from 220–250°) for an hour. Remove from the air bath, cool in a desiccator, and weigh again. Determine the per cent. of water of crystallization, assuming gypsum to have the formula $CaSO_4 + 2\ H_2O$. Compute as in the case of copper sulfate. Compute theoretical results and compare with your results. If the per cent. is too low it is quite probable that you must heat more to drive out the water.

NOTE.—It is quite necessary that every laboratory should have one good pair of balances. The instructor should give each student a lesson in weighing before any attempt is made to do quantitative work.

ULTIMATE ANALYSIS OF BARIUM CHLORID

WATER OF CRYSTALLIZATION.

I. Weigh out very carefully about two grams of barium chlorid and determine its water of crystallization as described before. Compute.

BARIUM.

II. Now weigh a clean small watch glass and record its weight. Next place about one gram of $BaCl_2$ on the glass and weigh again. Record the weight of the chlorid. Next wash it very carefully into a clean beaker and dissolve in distilled water. Heat almost to boiling, then precipitate the barium as barium sulfate with dilute sulfuric acid (one part concentrated acid to nine of water), stirring the solution with a glass rod during the precipitation. Heat for a minute or two, constantly stirring, then filter and wash with hot water. Be careful not to spill a single bit of the precipitate, as the least carelessness will make

great errors. When the precipitate is washed transfer the filter paper containing it to a crucible which has been carefully weighed. Always handle crucibles with pincers. Heat the crucible gently until the paper and precipitate are perfectly dry, then heat more strongly until both are reduced to a clean white powder. Cool in a desiccator and weigh. Tabulate your results and compute as follows:

EXAMPLE.

Weight of watch glass $+(BaCl_2+2H_2O)$	= 3.1075	grams.
" " " " (empty)	= 2.1098	"
" " $BaCl_2+2H_2O$	= 0.9977	"
" " crucible + $BaSO_4$	= 9.2811	"
" " empty crucible	= 8.3302	"
" " $BaSO_4$	= 0.9509	"
Per cent. of barium as calculated	= 56.14	
" " " " found	= 56.04	
Error	= 0.10	

COMPUTATIONS. (*Theoretical.*)

The molecular weight of $BaCl_2 + 2H_2O$: the atomic weight of Ba :: 100 : x or 244 : 137 :: 100 : x where x = the per cent of Ba.

$BaSO_4$: Ba :: .9509 : x, x = the actual weight of Ba in .9509 grams of $BaSO_4$ = .5590 grams; then the weight of $BaCl_2 + 2H_2O$: Ba :: 100 : y, where y = % Ba, or 0.9977 : .5590 :: 100 : y. y = 56.04.

CHLORIN.

III. Weigh out about one gram of $BaCl_2 + 2H_2O$ very carefully, as before. Wash into a clean beaker and dissolve in water. Heat and while hot precipitate the chlorin with silver nitrate. Silver chlorid is formed. Continue heating until the precipitate settles to the bottom

of the beaker. Filter and wash carefully with hot water. Place the filter paper and precipitate in a weighed crucible and allow to stand in a drying oven at 115° until perfectly dry. Now heat directly in a burner flame until the silver chlorid just begins to melt. Cool and weigh then compute the per cent of chlorin in $BaCl_2 + 2H_2O$.

EXAMPLE.

Watch glass + ($BaCl_2 + 2H_2O$)	= 3.0036	grams.
" " empty	= 2.1103	"
Weight of ($BaCl_2 + 2H_2O$)	= 0.8933	"
" " crucible + AgCl	= 8.3486	"
" " empty crucible	= 7.3187	"
" " AgCl	= 1.0299	"
Per cent. of chlorin calculated	= 29.10	"
" " " found	= 28.69	"
Error	= 0.41	"

Theoretical.—$BaCl_2 + 2H_2O : Cl_2 :: 100 : x$, where $x = \%$ Cl. $244 : 71 :: 100 : x$; $x = 29.10$.

FOUND.—$AgCl : Cl :: 1.0299 : x$. $x =$ actual weight Cl in 1.0299 grams AgCl.

$143 : 35.5 :: 1.0299 : x$. $x = .2556$ grams Cl in 1.0299 grams AgCl.

$BaCl_2 + 2H_2O : Cl_2 :: 100 : y$

$0.8933 : .2556 :: 100 : y$. $y = 28.69$.

To determine whether your results are correct, add the per cent of Cl, Ba, and H_2O and they should equal 100%.

SODIUM CHLORID.

Determine the amount of Cl in sodium chlorid following the above methods for chlorin.

COPPER SULFATE ($CuSO_4 + 5\ H_2O$).

I. Determine the water of crystallization of $CuSO_4$.

Note.—If this has been done correctly once before, the results of the first determination may be used.

II. Weigh out a crystal carefully, dissolve in water, heat and precipitate with barium chlorid. $BaSO_4$ will be formed. Dry and heat until reduced to a clean white powder. Weigh and calculate the SO_4 in $CuSo_4 + 5H_2O$. Tabulate results.

III. In the following experiment it is best to use a Rose crucible, *i. e.* one which is fitted with a cover and a porcelain delivery tube.

Weigh out a clean copper sulfate crystal. Heat gently to about 250° until all the water of crystallization is driven off. Then heat more strongly directly in the flame of a burner until the copper sulfate is changed to copper oxid, allowing a slow current of oxygen to pass into the crucible during the heating.

Calculate the amount of copper and add up your results to make 100%.

SILVER NITRATE.

Determine the amount of silver in a weighed sample of silver nitrate, using dilute hydrochloric acid to precipitate the silver. The process is a reversal of that already given for chlorin and the student will have no difficulty in following it.

DEFINITIONS.

I. **Acid.**—An acid may be defined as a compound of hydrogen and a non-metal usually in combination with oxygen. The acids containing oxygen are called oxacids. Examples are HNO_3, H_2SO_4, H_3PO_4, $HClO_3$, etc. An acid has a sour taste, reddens certain vegetable coloring mat-

ters such as litmus, and contains hydrogen which may be replaced by a metal.

II. **Non-metals.**—The non-metals which form acids are the halogens, sulfur, phosphorus, nitrogen, arsenic, etc.

III. **Salt.**—A salt is a compound in which the hydrogen of an acid has been replaced by a metal. As KNO_3, K_2SO_4, NaCl, etc.

IV. **Base.**—A base is an oxid or hydroxid of a metal. Bases dissolve in acids to form salts and water. As $KOH + HCl = KCl + H_2O$, etc.

Metals act like bases in some cases because they dissolve in acids to form salts, forming hydrogen instead of water. As $Zn + 2\ HCl = ZnCl_2 + 2\ H$, etc.

NOTE.—When a metal is treated with an acid, hydrogen is set free, but in many cases such as the solution of copper in sulfuric acid, or of any metal in nitric acid, the nascent hydrogen has a secondary action on the acid, and reduces it to form sulfur dioxid or nitric oxid, so that, although hydrogen is set free, it is disguised by the secondary products and is not the ultimate product of the reaction.

V. **An alkali** is a base that is soluble in water and turns litmus paper blue. As KOH, NH_4OH, etc.

VI. **An anhydrid** is a body which when combined with water will form an acid. As SO_2, SO_3, B_2O_3, As_2O_3, etc.

Oxacids whose names end in *ic* form salts ending in *ate*, while those containing less oxygen whose names end in *ous* form salts whose names end in *ite*. In general when a metal forms two classes of salts, those having a less amount of oxygen have names ending in *ous*, while the names of those having more oxygen end in *ic*. Examples are mercurous, mercuric, ferrous, ferric, etc., for adjectives, and mercurosum, mercuricum, etc., for nouns.

NUMBER OF GRAMS OF SOLIDS TO 500 cc. OF H_2O.

$AgNO_3$*	25	KCN	50	Na_2CO_3	50
$BaCl_2$	50	KCl	25	NaOH	60
$C_{12}H_{22}O_{11}$	sat	K_2CrO_4	25	NH_4Cl	60
$CaCl_2$	50	$K_2Cr_2O_7$	25	$(NH_4)_2CO_3$	125
$Ca(OH)_2$	sat	$K_4Fe(CN)_6$	40	$(NH_4)_2C_2O_4$	20
$CuCl_2$	25	$K_3Fe(CN)_6$	40	NH_4NO_3	50
$Cu(NO_3)_2$	25	KI	25	$(NH_4)_2SO_4$	5
$FeCl_3$	25	KNO_2	50	$Pb(C_2H_3O_2)_2$	50
$FeSO_4$*	50	KNO_3	50	$PbCl_2$	sat
$HNaCO_3$	50	KOH	60	$Pb(NO_3)$	50
$HNaPO_4$	50	$MgCl_2$	50	$SnCl_2$	40
$HgCl_2$	25	$MgSO_4$	50	$SrCl_2$	50
$HgNO_3$*	25	$MnCl_2$	25	$ZnCl_2$	25
KBr	25	NaCl	50		

Acid.—Whether concentrated, or dilute acids are to be used depends upon the kind of work in hand. The instructor should direct the pupil in this matter.

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* Do not filter $AgNO_3$, nor bring it in contact with organic matter. Ferrous sulphate should be prepared as needed. Make a mixture of 475 cc. of H_2O and 25 cc. of HNO_3 in preparing $HgNO_3$.

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